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G. Granier, B. Benezech, Jb. Porcher, Anne-Catherine Simon, N. Pepin, et al.. A methodology for evaluating the performance of software, using gamma spectrometry, for determining the isotopic composition of plutonium. ANIMMA 2009 - 1st International Conference on Advancements in Nuclear Instrumentation, Measurement Methods and their Applications, Commissariat à l'Energie Atomique (CEA); University of Provence; Belgian Nuclear Research Centre SCK-CEN, Jun 2009, Marseille, France. 10.1109/ANIMMA.2009.5503791 . cea-03994164

HAL Id: cea-03994164

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Submitted on 17 Feb 2023

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A Methodology for Evaluating the Performance of Software, using γ Spectrometry, for Determining the Isotopic Composition of Plutonium

G. Granier, B. Benezech, JB. Porcher, A-C. Simon, N. Pepin, E. Payan, C. Veyer

Abstract— This paper presents the progress of an ongoing study within Working Group N°27 of the CETAMA[1] regarding the performances of software used for determining the isotopic composition of plutonium and uranium, by means of γ spectrometry, in the presence of “disturbing” radioactive emitters and of various matrices. The “disturbing” radio-emitters are some minor actinides (^{242}Am , ^{243}Am , ^{237}Np , ^{243}Cm etc.), fission products (^{137}Cs , ^{125}Sb , ^{154}Eu etc.) and/or activation products (^{60}Co etc.). All these radionuclides can be found in waste from the nuclear industry. Matrices can also vary (metal, vinyl...). In some instances, these radionuclides and matrices are in high enough quantities to disturb the analysis by the software for determining the isotopic composition. This paper presents the outline and first achievements of this .

[1] The CETAMA (Commission d’Etablissement des Méthodes d’Analyse), part of the CEA, has served laboratories for analysis and monitoring facilities throughout the French nuclear sector. Working group N°27 brings together experts in neutron and gamma spectrometry measurements to identify suitable non destructive assay methods for waste packages of all shapes, all backgrounds and all levels of alpha or beta-gamma emitters.

Index Terms—waste, isotopic composition, γ spectrometry, interference

I. INTRODUCTION

THE French organization CETAMA (Commission d’Etablissement des Méthodes d’Analyse) must fulfill several missions for its customers in the nuclear industry:

- To propose scientific and technical developments aimed at improving the quality of chemical or nuclear measurements

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- To collect and harmonize analytical and measurement procedures
- To organise inter-laboratory comparisons

The CETAMA is organised around Working Groups aimed at facilitating the identification of needs, sharing lessons learned and disseminating knowledge in the field of analysis and measurement. The study described in this paper has been initiated and managed by the “*Measurement of VLA/LA and MA packages*” sub-group within the “*Non destructive analysis for waste and decommissioning*” Working Group (WG 27).

Isotopic composition (IC) determinations based on gamma measurements are commonly used to characterize nuclear waste when the physical (geometry, density) and radiological characteristics of the packages allow using the part of the gamma spectra situated between 50 and 200 keV. When the conditions are favorable, in most cases, gamma spectrometry can be used to quantify the activity of one or several actinides by using their emission lines between 150 and 600 keV.

An inter-laboratory round robin has been organized by the CETAMA from 2002 to 2004, during which five drums of simulated and actual solid waste were circulated between the 10 participating laboratories for the determination of activity for beta-gamma emitters, the determination of isotopic composition for U or Pu and the determination of activity and weight for each isotope of Pu (or U). This round-robin gave rise to numerous discussions and extensive analysis work on the lessons learned. During this discussions, the group of experts determined that it was necessary to obtain more information on potential difficulties and limits of use when applying the various isotopic composition software tools in the presence of some “interfering” radionuclides (actinides, fission or activation products), liable to be present in the waste.

Numerous studies have been performed in the past to determine the influence of measurement conditions and to estimate the precision of the various codes on simple configurations (samples). The M.G.A. code, developed by Lawrence Livermore national Laboratory, is currently the most widely used software for isotopic composition determination.

PC-F.R.A.M (Fixed-energy Response function Analysis with Multiple efficiency), developed at Los Alamos National Laboratory) is the second most used code in this field. A team of experts at CEA Saclay is developing a new code I.G.A. Those three codes allow analyzing gamma spectra recorded via germanium detectors. At the time of the study, very few information existed about the way those three codes dealt with “interfering” elements, especially when their concentration becomes significant.

A bibliographic study performed on those three codes showed that they operate on very different principles. The interfering elements of interest in common waste material are not always detected / quantified systematically, and may sometimes generate errors in the IC. This analysis thus confirms the need for a detailed and systematic analysis of the impact of these radionuclides.

II. ORIGINAL SPECIFICATIONS FOR THE STUDY

The original aim of the study was to determine the field of applicability of each IC determination software tool, when considering various types of package, radiological contents, measurement conditions and configuration, and equipment.

One method to perform this evaluation consists in obtaining spectra from waste packages with known isotopic compositions, and to submit these spectra to analysis by the various IC tools, in order to compare the results with the known composition.

In a first step, and for the sake of simplicity and exhaustiveness, it was proposed to generate these “test” spectra numerically. However it was soon concluded that computer simulation was not adapted, since, in the region of interest (around 100 keV), it was difficult to accurately model the interactions responsible for the continuous background in these energy ranges. One of the reasons for this is that the nuclear data used to describe these background phenomena are not well known (for absorption and diffusion mechanisms, specifically). In addition, the available nuclear and atomic data for the emission lines of actinides are sometimes known with significant uncertainties. The values found in the reference databases, and related to the peaks found in this region, differ both for the energy values and for the branching coefficients. Thus, by performing the evaluation on simulated spectra, it would not have been possible to discriminate between the actual performance of the IC software and the eventual differences in the databases used. It was then decided to perform the evaluation on spectra derived from experimental measurements.

A common project involving CEA, AREVA and IRSN was then launched, driven by the DTN.SMTM/LMN (CEA Cadarache) assisted by CETAMA. It was expected that the project would last for about 2 years.

The proposed method involved the following steps:

- Acquisition of “elementary” mono-isotopic or mono-elemental spectra on mock-up packages, with standard sources in well defined measurement conditions, to establish a common database.
- Computation of “virtual” spectra by summing these “elementary” spectra on each channel (in the same conditions).
- Testing of these virtual spectra (or TEST spectra) with the various software tools.

According to the original program, it was proposed to perform the acquisition of the “elementary” spectra on various package mock-ups, with each elementary standard source, at several positions in the package, and in conditions as reproducible as feasible.

A preliminary specification file was established to determine all the potential parameters of influence, and fix the test conditions. The preliminary matrix involved 4 to 10 Pu and U isotopic compositions, 4 to 8 interfering nuclides (actinides, fission products (FP) and activation products (AP)), and 4 packages with several possible source locations, for at least three types of detectors and varying measurement configurations. An example of a potential package configuration is presented on fig. 1.

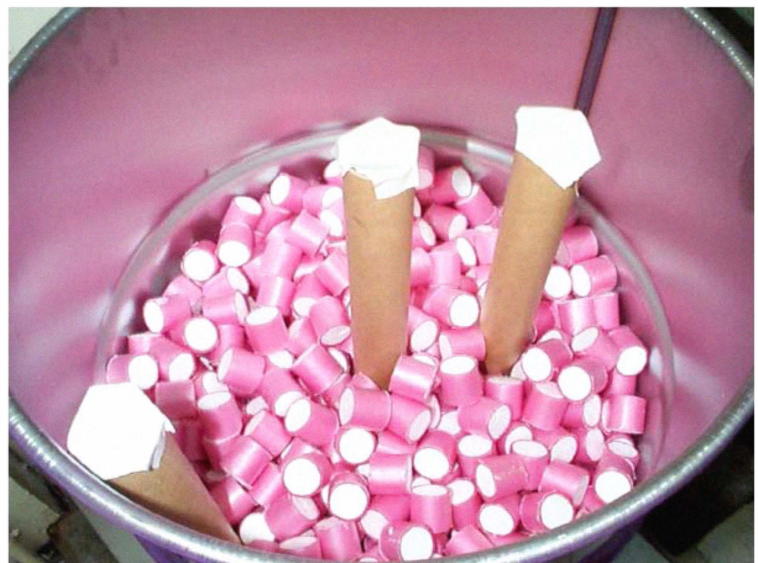


Fig. 1. Potential mock-up configuration

III. EVOLUTION OF THE SPECIFICATIONS

Soon in the process, it became evident that the time required to perform all the acquisitions would not allow completion of the project in the expected time frame. This resulted from the very large number of parameters that needed to be studied to establish the database (number of sources, number of detectors, number of matrices, and configurations...). In

addition, some of the sources required to explore the whole range of target “interfering” radionuclides were not available. The specifications were then adjusted to this new context.

A. Detectors

The group decided not to reduce the number of detectors, since it is necessary to provide information for a wide range of users.

B. Test Assemblies

The major proposed change consisted in performing all the required “elementary” acquisitions on a specific measurement bench instead of mock-up packages. This would allow significant simplifications in the process:

- Elimination of the package preparation step, and of the associated logistics to combine the packages and the sources and transport them to the measurement stations.
- Decrease of spatial requirements, thus allowing the implementation of three detectors with their electronics in a single room.
- Optimization of the counting time, since the distance between the detector and the source would not be constrained in the same way.
- Improved reproducibility
- Enlarged field of applicability (not limited to the geometry and configuration of the mock-up packages).

This change was accepted provided that the equivalence between the spectra obtained on the bench and on mock-up packages is validated.

Later, and since the bench allowed it, it was decided to add another kind of matrix to the study: a mixture of 50 wt% PVC and 50 wt% glass, with densities between 0.2 and 0.6 g.cm⁻³.

C. Radionuclides

No sources were identified for Co-60, Eu-154, Sb- 125, Cm-243 or Am-242m. The available sources were thus the following:

- 3 Pu sources containing between 80 and 97 % Pu-239
- Mono-isotope sources (28 years old) for Pu-238, Pu-240 and Pu-241
- Sources of interfering radionuclides: Am-241, Am-243, Np-237, Cs-137 and U-235.

Note: from the available Pu sources and the mono-isotopic sources, it may be difficult to simulate LWR reactor-grade plutonium, since for some isotopes the weighting coefficients would be quite high.

IV. VALIDATION OF THE PRINCIPLES

A. Feasibility of Operations on “Elementary” Spectra

The creation of virtual spectra by summing elementary spectra on each channel is theoretically feasible. The only physical limit is associated with the interactions (X-ray fluorescence) that may exist between the two sources when they are together, and that cannot be observed when measuring the sources independently. As a result, and since the IC software do not consider this type of rays, the comparison will only consider the field of gamma emissions.

In practice, the quality of the combination will depend on the stability of the measurement chain. The process must be verified experimentally. In order to validate this procedure, a preliminary experiment was performed with only one single collimated low energy Ge detector (2000 mm², 20 mm thickness). 4 sources of varying activities were selected, representing two known isotopic compositions. The conditions were selected to allow the comparison: effective recording times identical for all the spectra to be compared, subtraction of background for each spectrum, completely identical experimental conditions.

For direct summation of spectra, the sequence of acquisitions described in Table 1 was performed, both directly and through a PVC screen representing a waste matrix, for two sources with slightly differing IC.

TABLE 1: MEASUREMENT SEQUENCE TO TEST THE FEASIBILITY OF SUMMING ELEMENTARY SPECTRA

	Source	Active recording time	Distance between source and detector
Background 1	None	T1	:
Source 1	S1	T1	D
Background 2	None	T2 (> T1)	/
Source 2	S2	T2	D
Background 3	None	T2	/
Combined source 1	S1 and S2	T2 (T1 with both S1 and S2 and (T2-T1) with only S2)	D
Background 4	None	T2	/
Combined source 2	S1 and S2	T2 (T1 with both S1 and S2 and (T2-T1) with only S2)	D
Background 5	None	T2	/

The “net” spectra were obtained by subtracting for each channel the background from the corresponding spectrum. The “sum” was obtained by summing, for each channel, the net spectra from Source 1 and Source 2, as illustrated on fig.2. Two residuals were then calculated. The “statistical and experimental” residual was obtained by difference between the two “Combined source” spectra. The summation residual was obtained by difference between the “Combined spectrum 1” and the above-defined “sum”. The residuals were compared to each other and to the background in order to evaluate the significance of the variation between the sum and the combined spectra.

A validation was also performed for weighted sums, to allow simulating the spectrum of a mixture of components from the elementary spectra obtained with single sources. Weighting a net spectrum (i.e. multiplying the contents of each channel by

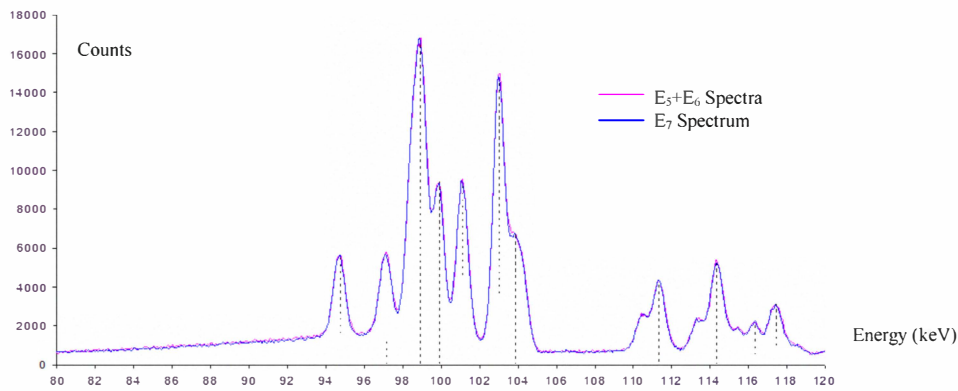


Fig. 2: example of direct summation, with a PVC screen

a constant) could reflect, for instance, a change in the activity of the source, or a change in the distance between the source and the detector. For the experimental study, it was decided to weight the elementary spectra from given sources according to counting time and the distance between the source and the detector. Again, the study was performed both with and without the presence of a PVC screen to simulate the waste matrix. The experimental sequence in both cases is given in Table 2.

TABLE 2:
MEASUREMENT SEQUENCE TO TEST THE FEASIBILITY OF A WEIGHTED SUM OF
ELEMENTARY SPECTRA

	Source	Active recording time	Distance between source the detector
Background 11	None	T5	:
Source 5	S1	T5	D1
Background 12	None	T6	/
Source 6	S2	T6	D2
Background 13	None	T7	/
Combined source 5	S1 and S2	T7	D3
Background 14	None	T8	/
Combined source 6	S1 and S2	T8	D3
Background 15	None	T8	/

The weighting is performed by adjusting the counting time (by the ratio of the counting times) and the distance (by the square of the distance ratio) to the selected common reference values.

These experiments allowed concluding that the rebuilding of a multi-source spectrum from elementary single source spectra is possible both with and without the PVC screen, as illustrated on figure 2, for instance. The quality of the virtual spectra is

nevertheless very dependant on rigorous performance of the acquisitions:

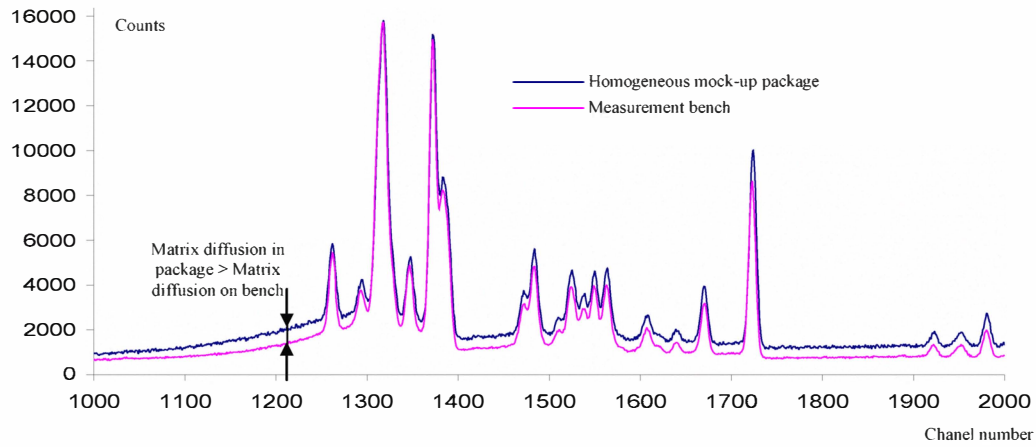
- The energy/channel relationships on the elementary spectra (and on the backgrounds) must be strictly identical. A specific preliminary tool was developed to recompute the contents of each channel from a spectrum according to the required energy/channel relationship, by first a compression and then a linearization of the spectrum.
- The distance between the source and the detector must be large when compared to the dimensions of the source or of the detecting surface.
- The acquisition time and distance between the detector and the source must be identical, or re-normalized for weighted sums.
- Anisotropic sources must be rotated to obtain an average spectrum and avoid any fluence rate variation

B. Equivalence of the Spectra Obtained on the Bench and on Mock-up Drums

In order to understand the effect of package configuration on the spectra, three series of tests were performed, using the same plutonium source:

- acquisition of the spectrum with the source situated in the centre of a simulated package filled with a homogeneous matrix
- acquisition of the spectrum with the source situated in the centre of a simulated package filled with a heterogeneous matrix, but with the same average density as the previous package
- acquisition of a “virtual” spectrum on the bench.

The homogeneous package was a 118 L steel drum filled with small cylinders of polystyrene coated with PVC, in order to obtain an average density of 0.29 g.cm^{-2} (see fig. 1 for instance). The heterogeneous package was obtained by inserting in the “homogeneous” matrix one block of solid PVC and empty PVC containers, so that the average density of those two insertions result in the same apparent density of 0.29 g.cm^{-2} . Those two “heterogeneities” were positioned along the measurement axis to maximize the impact.

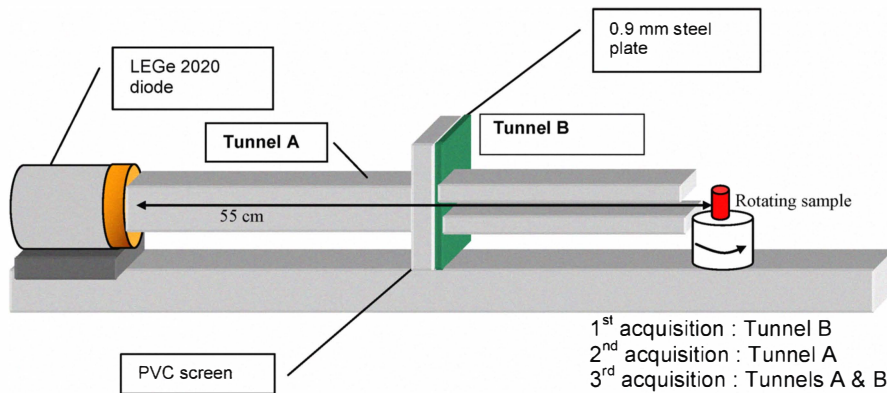


Fig; 3. Comparison of spectra from a homogeneous mock-up package and from the bench: effect of matrix diffusion

The two packages were measured with and without the Pu source, in rotation, with the detector positioned at mid-height. The bench measurement was performed by positioning the source (in rotation) in front of a shielded detector (to minimize the effects from the surroundings) and through a screen

homogeneous matrix of the package were higher than those in the PVC screen used on the bench (see fig. 3 for instance).

A systematic parametric study was then performed to adjust the bench set-up and improve the representativeness of the simulation (the aim was to obtain diffusion ratios¹ similar to



composed of 0.9 mm steel (to simulate the drum) and with a thickness of PVC calculated to provide the same attenuation as the matrix in the drum. The comparison of the two spectra from the “homogeneous” package and from the bench assembly put into light the fact that the diffusion effects in the

those obtained with the packages). It was found that the impacts of the distance between the source and the screen or of

¹ the diffusion ratio is defined as the ratio of the area below the baseline immediately before the peak and the area below the peak, determined over equal widths of energy intervals.

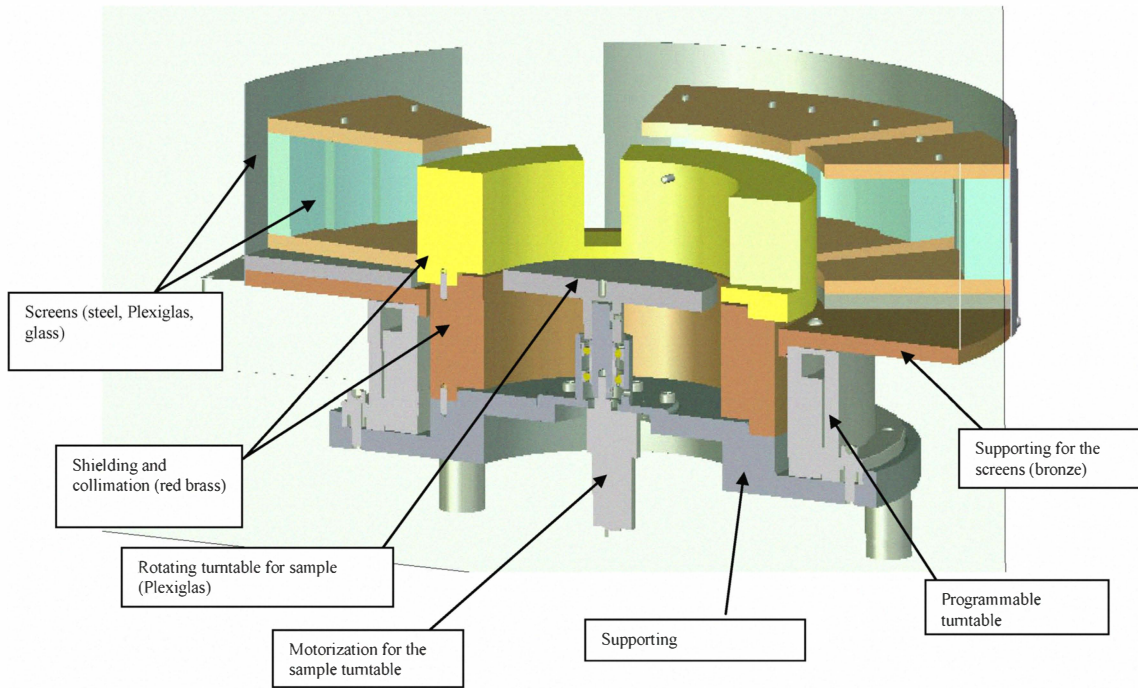


Figure 6: General view of the SCHEMAS bench

the size of the screen were limited. The most significant improvement was obtained by adding “diffusion tunnels” to the system, between the detector and the screen and between the screen and the source, wide enough to not interfere with the detection solid angle. See fig. 4 and 5.

V. DESIGN AND CONSTRUCTION OF THE SCHEMAS BENCH

The SCHEMAS (*Simulateur de Colis de déchets HETerogènes pour la Mesure d’Actinides par Spectrométrie gamma*) modular bench has been designed according to the conclusions of the above studies. It has been designed to simulate the interactions between radiations and various materials that can be found in real waste drums by incorporating mobile screens with varying compositions and thicknesses, and able to simulate homogeneous as well as heterogeneous matrices. The bench is based on an existing programmable precision turntable, and can be connected to three acquisition systems at 120° from each other. See figure 6 to 8. The source is in the

centre, on a rotating support. Around the source, a collimation and shielding block is positioned, to provide the three measurement windows, ensure biological shielding and limit unwanted diffusion phenomena. This block is made of “red brass” to limit unwanted X-ray fluorescence phenomena. The various matrices and their combinations are simulated by three series of 10 screens each (including steel plate, glass, Plexiglas...) that can be combined in three different zones according to the required setting. The thickness of the various screens is computed to simulate the attenuation of a reference drum, filled with homogeneous repartitions of masses, materials and source. See table 3. These 3 screen zones are positioned on the programmable turntable and separated by empty zones which allow performing the measurement of the bare source. Inside the bench, the diffusion tunnels are composed of two Plexiglas horizontal plates above and below the source.

The bench was constructed in May 2007 and set-up at the LMN at Cadarache.

TABLE 3: EQUIVALENT SCREEN THICKNESSES TO SIMULATE VARIOUS PACKAGE CONFIGURATIONS

Matrix in the drum	Average matrix density in drum (g.cm ⁻³)	Equivalent thickness PVC in mm (ρ = 1.19 g.cm ⁻³)	Equivalent thickness Fe in mm (ρ = 1.19 g.cm ⁻³)	Equivalent thickness glass in mm (ρ = 1.19 g.cm ⁻³)
100 % PVC	0.2	48.0		
	0.4	65.0		
50 wt% Fe, 50 wt% PVC	0.3	23.12	3.5	
	0.7	39.63	6.0	
50 wt% glass, 50 wt% PVC	0.3	28.0		13.1
	0.5	59.0		21.2

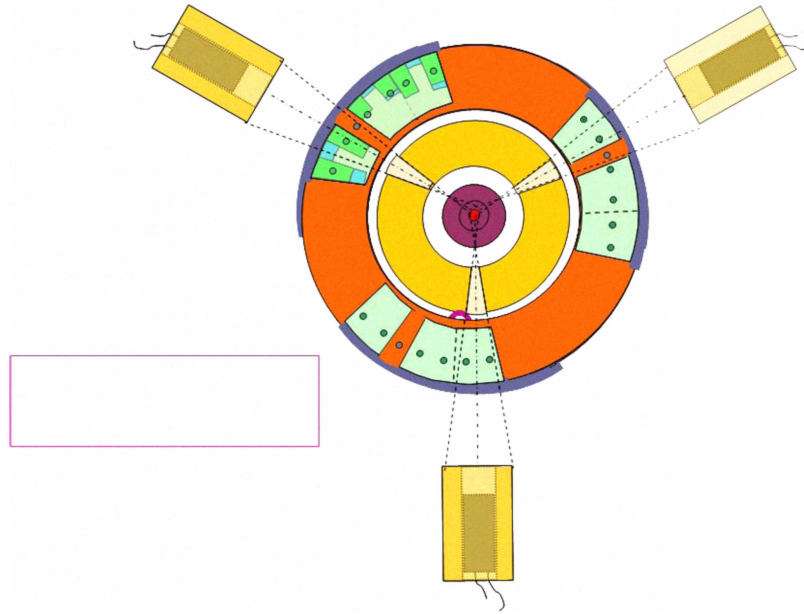


Fig. 7. General disposition of the screen sequence

VI. PROCEDURES FOR ACQUISITION AND CREATION OF THE TEST SPECTRA

In addition to the bench tool described above, the study has allowed setting up procedures and parameters to perform the acquisitions and create the “virtual spectra” that will be analyzed by the IC software tools.

A. Acquisition of the Elementary Spectra

The criterion required by MGA to attenuate the 59.54 keV peak from Am-241 is the most conservative. It has thus been decided to apply this criterion for all the measurements. The thickness of the absorbing screen must be such that the 59.54 keV peak is attenuated to a level comparable to those present around 100 keV. The screen is made of tin (Sn).

In order to minimize the impact of any heterogeneity, the sample is systematically rotated at a constant speed. The number of rotations during the acquisition must be an integer number, or, alternatively, be so high that the effect of the non completed last rotation is not significant. The counting rate

must be such that degradations associated with high counting rates (losses, piling, and decrease of the resolution) can be neglected. This is ensured by setting the distance between the source and the detector, for each source and each detector. The distances between the sample and the detector may vary between recordings, provided that the principles of weighting defined above are complied with.

The electronics should be able to be used over 16000 channels or more. The gain should be set according to the counting rate, in order to obtain an energy/channel relationship around 0.0375 keV/channel (for good counting rates) or 0.075 keV/channel (for low counting rates). If the sample contains known radionuclides that emit at least three lines spread over the required energy range, the gain is set directly with the sample. For samples that do not allow this, or for a background, a standard source must be used and three recordings must be performed (standard/sample or background/standard).

The counting time must comply with the requirements for the various software tools to be tested. The most conservative one



Fig. 8. The SCHEMAS bench

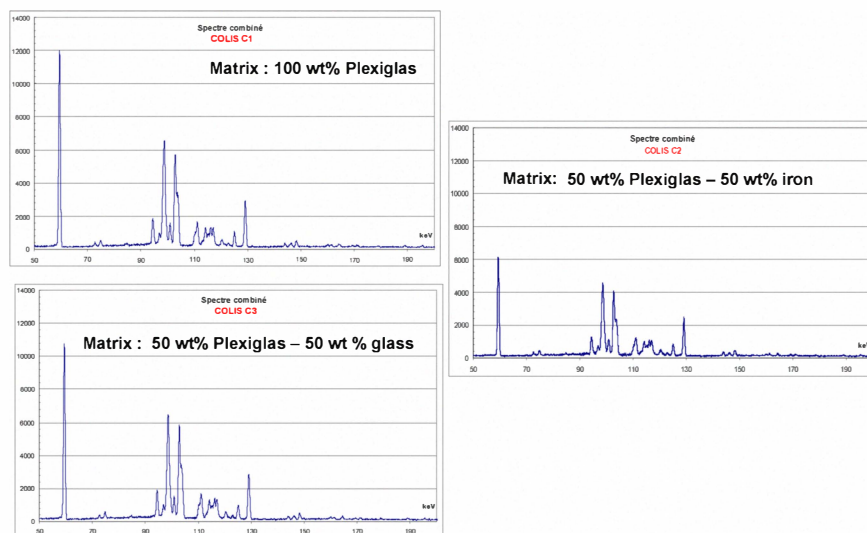


Fig. 9. effect of the matrix on the “virtual” spectrum of 10 g of GCR-type Pu in 3 virtual 200 L waste drums

is MGA which requires at least 10^6 counts for a plutonium sample. The criterion set for the study is 1.5×10^6 counts in the spectrum. For samples that do not contain Pu (pure interfering elements, for instance), the counting statistics associated to the various peaks must allow unambiguous interpretation.

B. Processing of the Elementary Spectra

For known spectra, the energy/channel correction is directly performed on 3 intense peaks covering the whole energy range of interest, by using the values found in a database (JEFF 3.1 for instance). The experimental data are then fit using a second order relationship and linearized using the tool described above. For spectra which require the use of a standard source before and after the measurement, it is essential to make sure that the two spectra from the standard source display the same energy/channel relationship, in order to use this relationship for the experimental spectrum. This verification is performed using three intense peaks covering the energy range of interest. The background recordings are spectra that require the use of standard sources. They will be processed accordingly, before subtraction from the (processed) sample spectrum.

C. Creation of the “Virtual” Spectra to be Tested with the IC Software Tools

The virtual (or TEST) spectra may be created by weighting directly the spectra according to the activities or, if the activities are not known, by indicating a proportion between the area of two peaks: a plutonium (preferably 239) peak and a peak from the pollutant. Specific attention is needed for the standardization aspects.

D. Compression or truncating of the spectra

The TEST spectra may then need to be compressed and/or truncated according to the requirements of the IC software tools, and converted to the adequate format.

VII. STATUS OF THE STUDY AND FIRST RESULTS

At the present date a simplified campaign is being performed, which allows testing the methodology, and the bench, and identifying the remaining integration issues, in order to finalize the software needed to operate SCHEMAS and process the acquisition results.

Acquisitions have been performed using one planar, small volume detector, with the following sources: one Pu sample with known isotopic composition (GCR-type), and three pure actinide sources (Am-243, Np-237, and Am-241). An acquisition with one Pu source and one Np237 source together has also been performed. Three types of matrices have been tested: C1 (100% Plexiglas, average density 0.3 g.cm^{-3}), C2 (50 wt% Plexiglas and 50 wt% metal - average density 0.3 g.cm^{-3}) and C3 (50 wt% Plexiglas and 50 wt% glass - average density 0.3 g.cm^{-3}). The acquisition time was between 8 and 12 hours per source. Figures 9 and 10 below illustrate the effect of the matrix on the measured spectrum and the effect of a pollutant on a recombined spectrum. Figure 9 illustrates the description by this method of the deformation (peaks and continuous baseline) of a spectrum corresponding to 10 g of GCR-type plutonium in a 200 l drum with the three different matrices (same apparent density). On figure 10, the effect of increasing amounts of Am-243 on the spectrum is computed for a C1-type package with 10 g of GCR-type Pu. It can be seen that, when the amount of Am-243 increases, additional peaks appear, with intensities that can become similar to those from the plutonium. One can also observe a change in the shape of the peaks and a progressive rising of the baseline. These effects may have an impact on the interpretation of the spectra by the IC software tools.

The raw test spectra have now been transferred to the laboratories in charge of testing and validating the method, who are now processing the spectra and subjecting them to the various IC software tools. Some minor difficulties are now

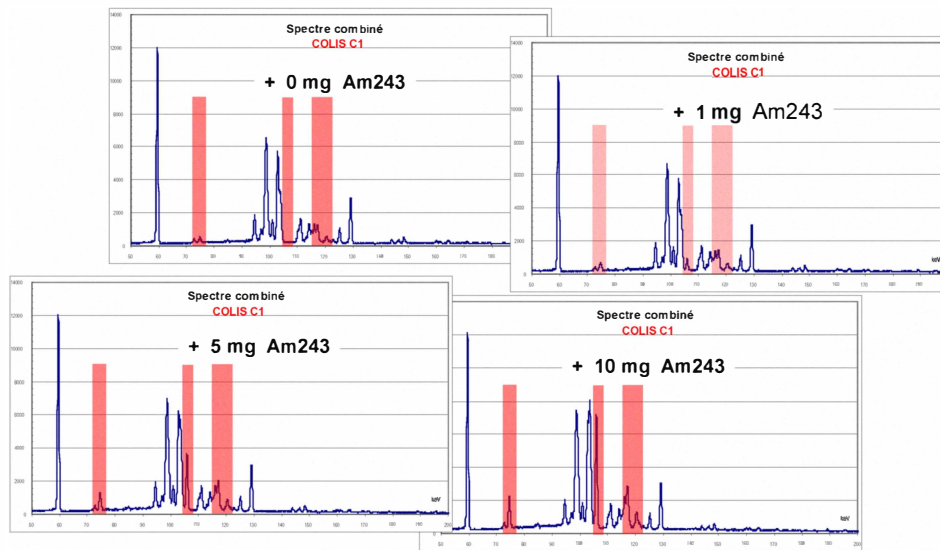


Figure 10: effect of increasing Am-243 amounts on the “virtual” spectrum of 10 g of GCR-type Pu in a 200 L drum

being corrected: in some instances, data formatting for the various software tools under study was not consistent.

radionuclides and matrix compositions, using the three software tools.

An example of the effect of these pollutants on the results given by an IC software tool is given in figure 11 below. On this figure, the ratios between the mass fractions of each isotope computed by IGA over the actual mass fraction of this isotope in the starting spectrum are plotted versus increasing Np-237 contents (which can be found in the label of each abscissa). It can be observed that, for this specific Pu IC and configuration, the addition of 0.5 % or more of Np-237 in the mix leads to serious discrepancies, especially for Pu-240 which, here, can be overestimated or underestimated by a factor of more than 2. Similar systematic testing is being performed for various isotopic compositions, interfering

VIII. CONCLUSION

A tool and procedures to generate complex gamma test spectra from individually measured elementary spectra have now been developed and validated. The system needs some additional work to become fully integrated: specifically, the software which allows operating the bench and processing the measurements needs to be developed on the basis of the procedure described above, and accounting for the lessons learned in the laboratories during this first exercise. This powerful tool could be used to generate test spectra and study a large number of parameters and configurations that could

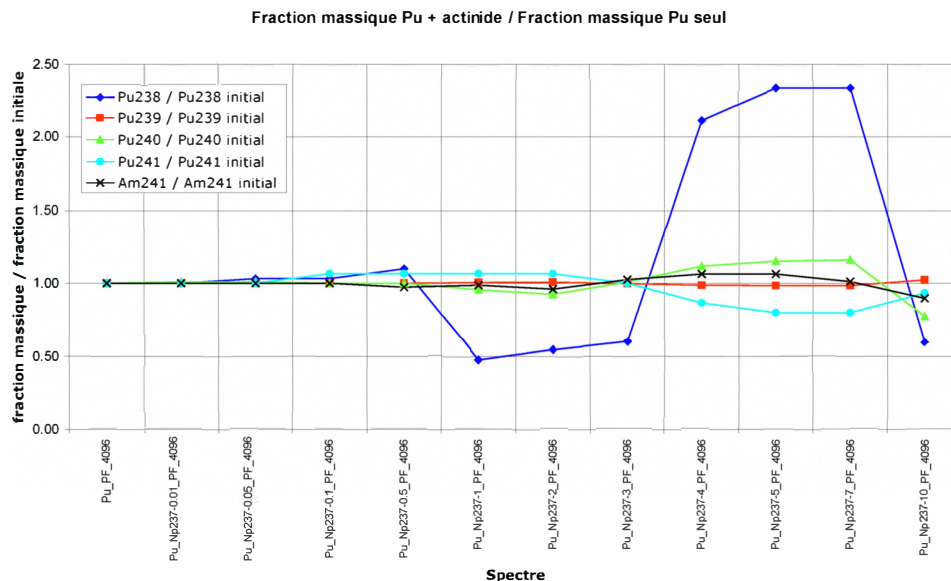


Figure 11: effect of increasing Np-237 contents on the results of IC determination using the IGA software tool. The Pu isotopic composition, measurement conditions, and package configuration are fixed

have an impact on waste characterization based on gamma spectrometry. These data would be based on actual experimental data in an energy range where numerical simulation is not easy or very reliable. The system is cheaper, simpler, more reproducible than experiments performed on actual packages or mock-ups, and allows testing an increased number of parameters, with the following advantages:

- Elimination of the package preparation step, and of the associated logistics to combine the packages and the sources and transport them to the measurement stations.
- Decrease of spatial requirements, thus allowing the implementation of three detectors with their electronics in a single room.
- Optimization of the counting time, since the distance between the detector and the source would not be constrained in the same way.
- Improved reproducibility

- Enlarged field of applicability (not limited to the geometry and configuration of the mock-up packages or to the available isotopic mixtures).

For the original objective of the study, which was to determine the field of application of the various software tools for Isotopic Composition in the context of waste management, a preliminary campaign, based on a reduced data base, has now been started. The preliminary results confirm that the methodology provides a means of testing the IC software tools as expected, by identifying levels of interfering radionuclides liable to induce discrepancies. At the end of this exercise, it is expected that the comprehensive results will allow guidelines to be set, help the users perform reliable gamma spectrometry-based Isotopic Composition determinations on waste packages.