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# Characterization of Old Nuclear Waste Packages

## Coupling Photon Activation Analysis and Complementary Non-Destructive Techniques

Frédéric Carrel\*, Bruno Charbonnier, Romain Coulon, Frédéric Lainé, Stéphane Normand, Corine Salmon, Adrien Sari

**Abstract**—Radiological characterization of nuclear waste packages is an industrial issue in order to select the best mode of storage. The characterization becomes crucial particularly for waste packages produced at the beginning of the French nuclear industry. For the latter, available information is often incomplete and some key parameters are sometimes missing (content of the package, alpha-activity, fissile mass...) In this case, the use of non-destructive methods, both passive and active, is an appropriate solution to characterize nuclear waste packages and to obtain all the information of interest. In this article, we present the results of a complete characterization carried out on the TE 1060 block, which is a nuclear waste package produced during the 1960's in Saclay. This characterization is part of the DEMSAC (Dismantling of Saclay's facilities) project (ICPE part). It has been carried out in the SAPHIR facility, located in Saclay and housing a linear electron accelerator. This work enables to show the great interest of active methods (photon activation analysis and high-energy imaging) as soon as passive techniques encounter severe limitations.

**Index Terms**—Photofission, SAPHIR, delayed neutrons, delayed gamma-rays, nuclear waste packages, LINAC.

## I. INTRODUCTION

Radiological characterization of nuclear waste packages is one of the most challenging aspects of the nuclear industry. Accurate information relative to the content of the package is required to select the best mode of storage. Non-destructive passive and active methods have been developed for decades and are powerful tools to obtain all the information of interest without opening the package, which enables to preserve its integrity [1, 2, 3].

If the content of nuclear waste packages currently produced is quite well-known, due to the improvement of measurement techniques over the years, the characterization of old nuclear waste packages, produced at the beginning of the French nuclear industry, is not so complete and some key parameters are sometimes missing (content of the package, alpha-activity,

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fissile mass...) For this reason, a fine characterization is required to reevaluate the content of the package. In this case, the coupling between several non-destructive techniques often becomes necessary, a single method being not enough to obtain all the information of interest.

In this article, we present the results of a complete characterization carried out on the TE 1060 block, which is a nuclear package produced during the 1960's. This characterization has been carried out in the SAPHIR facility, located in Saclay and housing a linear electron accelerator. First, we will remind the main characteristics of the TE 1060 block. Then, we will show experimental results obtained using traditional non-destructive passive techniques (gamma-ray spectrometry, passive neutron counting). In a third part, we will focus on high-energy radiography measurements enabling to obtain information on the content of the package. The last part will be dedicated to the presentation of results based on the photon activation analysis principle.

## II. CHARACTERISTICS OF THE TE 1060 BLOCK

The TE 1060 block is an historical nuclear waste produced in the Saclay site (see Fig. 1). It has been used for decades as a radiological protection in the Saturne synchrotron [4].



Fig. 1. View of the TE 1060 block.

This block is a 4.4 tons waste package containing a 220 liter barrel surrounded by a concrete wall (concrete thickness equal to 31.4 cm, considering that the barrel is positioned at the centre of the block). The TE 1060 package has the following external dimensions: height of 150 cm, width of 120 cm,

length of 120 cm. The 220 liter barrel contains old nuclear waste with uranium. The uranium isotopic composition is unknown for this type of waste. The density of the waste has been arbitrarily fixed at 1. In the frame of this characterization, an important point concerns the lack of information related to the concrete chemical composition (density, hydrogen content) which will have a significant impact on the quantification step.

A previous characterization of this block has been carried out by another team using passive gamma-ray techniques. However, the experimental configuration was not efficient enough to detect uranium contained inside the block.

### III. CHARACTERIZATION OF THE TE 1060 BLOCK USING PASSIVE NON-DESTRUCTIVE TECHNIQUES

First of all, we carried out passive gamma-ray spectrometry acquisitions using an experimental configuration more performing than the one previously used. Improvements are related to the type of detector (p-type ORTEC GEM40P-PLUS HPGe detector, relative efficiency equal to 40 %, connected to an ORTEC DSPECPro digital gamma-ray spectrometer instead of a 25 % relative efficiency HPGe detector), measurement distance (5 cm instead of 70 cm) and acquisition time (always higher than 12 hours instead of 1 hour for previous tests). Due to its high energy, the peak emitted at 1001.0 keV by  $^{234m}\text{Pa}$  was used to monitor the  $^{238}\text{U}$  activity. Fig. 2 illustrates the experimental configuration for the gamma-ray spectrometry acquisitions. Fig. 3 shows an example of spectra obtained during these measurements.

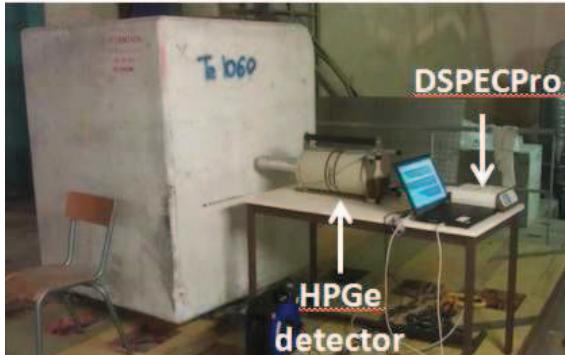


Fig. 2. Experimental configuration for the gamma-ray spectrometry acquisitions.

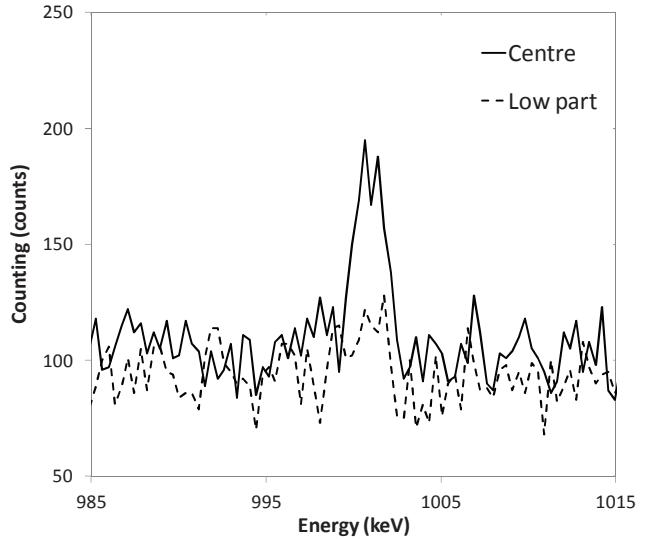


Fig. 3. Gamma-ray spectra obtained on the TE 1060 block. Two spectra have been superimposed, corresponding to measurements carried out at the centre of the block (full curve) or in the low part of the latter (dotted curve). A lead collimator has been used for these specific measurements.

The gamma-ray measurement carried out in the low part of the TE 1060 block confirmed that uranium traces (ppm quantities), often present in concrete, have no significant impact on our results (no detectable signal at 1001.0 keV). For this reason, we can consider that the peak detected at the centre of the package is due to the presence of  $^{238}\text{U}$  contained inside the 220 liter drum.

In order to evaluate the activity repartition inside the 220 liter drum, measurements have been carried out at the centre of the four sides of the block without using a lead collimator. The latter reveal an excess of signal on one of the sides, as it can be seen in Table 1.

TABLE 1  
COUNTING RATE (CPS) IN THE 1001.0 KEV PEAK FOR GAMMA-RAY SPECTROMETRY MEASUREMENTS CARRIED OUT AT THE CENTRE OF THE FOUR SIDES OF THE BLOCK. THE STATISTICAL RELATIVE UNCERTAINTY ON THE COUNTING RATES IS GIVEN AT ONE SIGMA.

	<b>Side A</b>	<b>Side B</b>	<b>Side C</b>	<b>Side D</b>
<b>1001 keV</b> $(\times 10^{-3} \text{ cps})$	9.88 (15.8 %)	5.73 (31.5 %)	10.27 (8.2 %)	12.72 (11.8 %)

Additional measurements have been carried out, in order to obtain a first evaluation on the localization of actinides. The latter were focused on the side D of the block, presenting the highest activity. Two different experimental configurations have been considered: with and without a lead shielding (opening area of 10 cm by 10 cm). Results are presented in Table 2.

TABLE 2  
COUNTING RATE (CPS) IN THE 1001 KEV PEAK FOR GAMMA-RAY SPECTROMETRY MEASUREMENTS CARRIED OUT AT DIFFERENT ALTITUDES ON SIDE D. THE STATISTICAL RELATIVE UNCERTAINTY ON THE COUNTING RATES IS GIVEN AT ONE SIGMA. ALTITUDE VALUES HAVE BEEN MEASURED FROM THE LOW PART OF THE TE 1060 BLOCK.

Measurement altitude	With lead collimator	Without lead collimator
H=44 cm	5.00 (16.9 %)	-
H=64 cm	5.78 (8.8 %)	14.63 (13.1 %)
H=84 cm	3.83 (23.1 %)	10.61 (8.6 %)
H=104 cm	-	5.22 (16.9 %)

Experimental results showed an excess of signal at 64 cm in both experimental configurations. It is important to emphasize that the counting time was quite long for each acquisition (exceeding 12 hours), due to the low intensity of the gamma-ray emission at 1001.0 keV (0.835 %) and because of the low intrinsic activity of  $^{238}\text{U}$  (12 kBq.g $^{-1}$ ). For this reason, the number of measurements and their precision still remain limited. These results were not sufficient to obtain a fine localization of nuclear material.

A specific long gamma-ray measurement (several days) has been carried out in order to determine the uranium isotopic composition using the IGA code [5]. However, due to the reduced number of peaks available in the spectrum, the analysis was unsuccessful.

At this step of the analysis, a first quantification has been carried out, assuming two configurations for the actinide repartition: centered inside the package or fully spread inside the package. The mean net signal obtained on the four sides of the block was considered in this calculation. The efficiency at 1001.0 keV has been evaluated using the Monte Carlo MCNPX code [6, 7]. The rebuilt  $^{238}\text{U}$  mass is equal to 495 g  $\pm$  151 g in the first case and to 464 g  $\pm$  142 g in the second case (uncertainty on simulated gamma detection efficiency equal to 5.6 % taken into account in this calculation).

In order to complete this passive characterization, additional passive neutron measurements have been carried out at different altitudes using the neutron counting system available in the SAPHIR facility (use of six  $^3\text{He}$  counters embedded in  $\text{CH}_2$  blocks wrapped with a 1 mm-thick cadmium foil). No passive neutron signal could be detected, which is consistent with the expected content of the block (absence of plutonium, which is often a significant source of passive neutron emission). The  $^{240}\text{Pu}$  detection limit has been evaluated to be equal to 92 mg in the frame of these measurements.

#### IV. CHARACTERIZATION OF THE TE 1060 BLOCK USING ACTIVE NON-DESTRUCTIVE TECHNIQUES

Non-destructive active measurements dedicated to the characterization of the TE 1060 block have been carried out using the linear accelerator (LINAC) of the SAPHIR facility. Main characteristics of the latter are the following: 5 mm tungsten target used to convert electrons into high-energy photons, peak current equal to 100 mA, pulse duration of 2.5  $\mu\text{s}$ , irradiation frequency of 25 Hz or 50 Hz and mean electron energy contained between 10.9 MeV and 18 MeV for these experiments.

##### A. High-energy Radiography Results

High-energy imaging is one of the most important technique in the frame of the characterization of nuclear waste packages in order to obtain information on their content [8]. The principle is identical to those of traditional X-ray radiography, except that the X-ray tube is replaced by a LINAC. To our knowledge, it was the first time that a high-energy measurement was carried out on this type of large and heavy package. The latter was possible using the mechanical conveyor of the SAPHIR facility, able to support packages up to 6 tons. For this experiment, we used a FIMEL Electronic Portal Imaging Device (EPID) which is an imager traditionally used for medical applications (see Fig. 4). This system uses a scintillating screen (500  $\mu\text{m}$  thickness, gadolinium), associated with a CCD camera, and enables to obtain a result directly at the end of the acquisition. The active area of the imager is equal to 30 cm  $\times$  30 cm, which implies that a complete image of the package requires several acquisitions (80 s each) and a reconstruction work to obtain the final result.



Fig. 4. The FIMEL EPID used for high-energy experiments carried out in the SAPHIR facility.

Fig. 5 presents an example of high-energy imaging results obtained for this type of package. The 220 liter drum can be clearly distinguished and its low-density content, in comparison with the concrete density, can be confirmed.

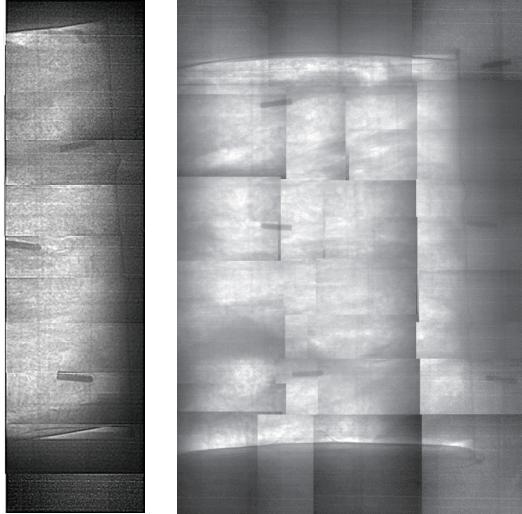


Fig. 5. On the left : partial view of the 220 liter drum (specific form of the metallic part of this type of package can be distinguished). On the right: global view of the package. Metallic uranium barrels have been used as markers on images.

### B. Characterization by Photon Activation Analysis

Photon activation analysis aims at detecting the presence of  $^{238}\text{U}$  contained inside the TE 1060 block. For these measurements, the experimental configuration is the following: use of twelve  $^3\text{He}$  counters (150NH100 model) spread into four detection blocks (two blocs on each side of the package). The energy of electrons was equal to 14 MeV, below the activation energy threshold of  $^{18}\text{O} (\gamma, p) ^{17}\text{N}$ , which can be a significant source of active neutron background. The first step of our analysis concerns the localization of actinides inside the package using the altitude scan method (irradiation of the package at different altitudes and detection of delayed neutron signal between irradiation pulses). The measurement duration for each acquisition was equal to 10 min. This analysis was carried out for each side of the block. Fig. 6 presents experimental results which have been obtained following this analysis. A clear hot spot can be identified at the altitude of 64 cm and the most intense signal is obtained on side D, which confirms previous gamma-ray spectrometry results.

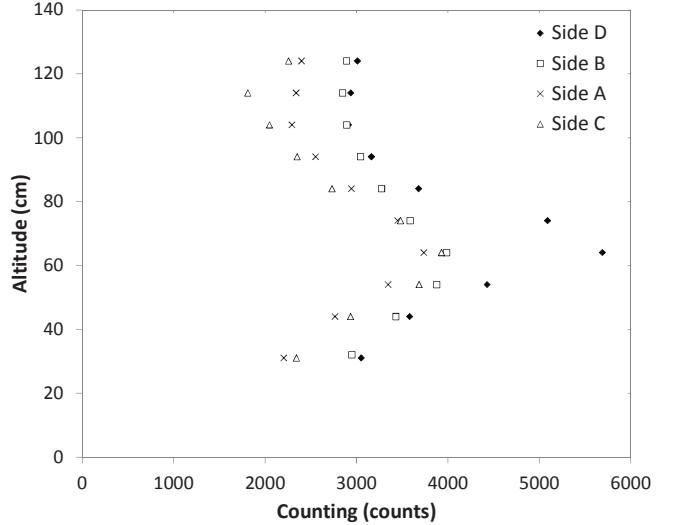


Fig. 6. Results of the altitude scan analysis (delayed neutron counting versus measurement altitude) for the four sides of the TE 1060 block.

Additional measurements have been carried out for an irradiation energy of 10.9 MeV (other parameters, including irradiation duration, were unchanged). Experimental results obtained for this specific measurement are exposed in Fig. 7. The hot spot at 64 cm is always clearly visible which indicates that the characterization of this type of blocks could be done using a lower energy transportable LINAC (for instance, a MiniLINATRON or a Varian LINAC M9).

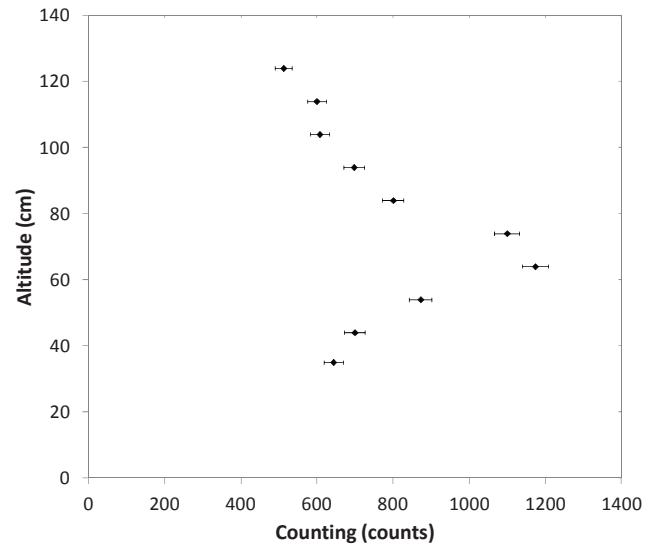


Fig. 7. Results of the altitude scan analysis (delayed neutron counting versus measurement altitude). The measurement was carried out on the side D of the block and for an irradiation energy of 10.9 MeV.

A last measurement has been carried out in order to localize the actinide mass, detecting high-energy delayed gamma-rays for the first time on a real nuclear waste package. This measurement is based on a counting of delayed gamma-rays emitted on a high-energy range (energy threshold set to 3 MeV), which enables to minimize the active gamma background. The feasibility of this approach was demonstrated

on a 870 L mock-up package but was never tested on a real nuclear waste package [9]. For this measurement, the durations of the irradiation, cooling and counting steps were respectively equal to 150 s, 10 s and 150 s. Only one BGO detector (3 inches×3 inches) was used. Fig. 8 presents experimental results obtained at the end of this analysis, which confirm the presence of a hot spot at the altitude of 64 cm.

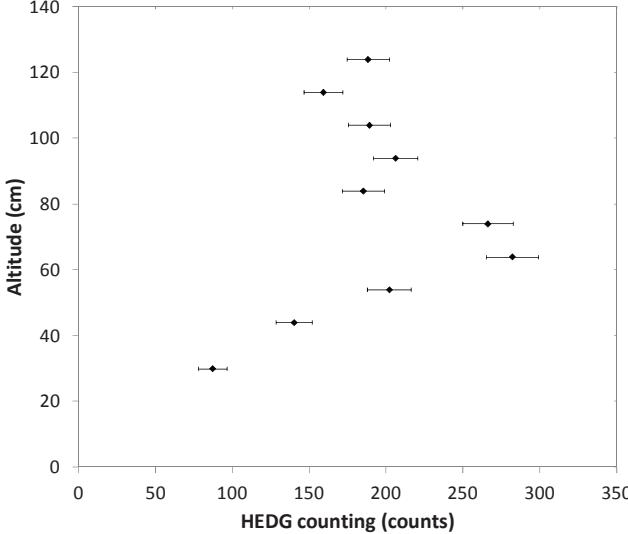


Fig. 8. Results of the altitude scan analysis based on the high-energy delayed gamma-ray (HEDG) counting. The measurement was carried out on the side D of the TE1060 block.

Due to time constraints, the analysis of high-energy delayed gamma-ray signal was not used in a quantitative purpose. It is important to emphasize that this work would be possible, thanks to an important work carried out during the last years by CEA IRFU and dedicated to the classification of high-energy gamma-ray emitters [10].

In order to complete this localization work, a fine analysis has been carried out, in order to rebuild the actinide mass present in each slice of the package. The considered approach was the following: the package has been split-up into eight regions of interest (height of 9.75 cm, diameter equal to 57.2 cm), enabling to fully cover the content of the 220 liter drum (78 cm height). The photofission sensitivity method has been calculated for each slice and for each irradiation position (ten acquisition points considered in the analysis). The entire measurement data set can then be formulated as an inverse problem, under the following form:

$$S = H \ M,$$

with

$$S = \begin{pmatrix} S^{pos 1} \\ \dots \\ S^{pos n} \end{pmatrix}, M = \begin{pmatrix} M_1 \\ \dots \\ M_p \end{pmatrix},$$

$$H = \begin{pmatrix} T_{Z_1}^{pos 1} \times \epsilon_{Z_1}^{pos 1} & \dots & T_{Z_p}^{pos 1} \times \epsilon_{Z_p}^{pos 1} \\ \dots & \dots & \dots \\ T_{Z_1}^{pos n} \times \epsilon_{Z_1}^{pos n} & \dots & T_{Z_p}^{pos n} \times \epsilon_{Z_p}^{pos n} \end{pmatrix},$$

$S^{pos 1}$  being the net delayed neutron signal measured in the irradiation position 1,  $T_{Z_p}^{pos n} \times \epsilon_{Z_p}^{pos n}$  corresponding to the photofission sensitivity for the irradiation position  $n$  and for the  $p$  slice and  $M_p$  being the actinide mass in the  $p$  slice. The inverse problem is solved using an Expectation Maximization (EM) algorithm [11]. The result of this analysis is presented in Fig. 9, which confirms that more than 98 % of the actinide mass is concentrated in regions 3 and 4. Fig. 10 compares delayed neutron net signal obtained during the altitude scan and the rebuilt signal using the actinide repartition given by the EM algorithm. The agreement is quite satisfying between experimental and rebuilt data, which confirms the validity of this processing. It is important to emphasize that this inverse problem approach could be used for different types of non-destructive techniques (for instance, passive gamma-ray spectrometry or passive neutron counting).

Z <sub>8</sub>	0.0%
Z <sub>7</sub>	0.0%
Z <sub>6</sub>	0.0%
Z <sub>5</sub>	0.0%
Z <sub>4</sub>	37.5%
Z <sub>3</sub>	61.0%
Z <sub>2</sub>	1.5%
Z <sub>1</sub>	0.0%

↑  
H<sub>T</sub>=78 cm  
↓  
H<sub>z</sub>=9.75 cm

Fig. 9. Results given by the EM algorithm (relative actinide mass present in each slice of the package). The origin of the first slice corresponds to the low part of the 220 liter drum, meaning an altitude of 36 cm regarding the global height of the TE 1060 block.

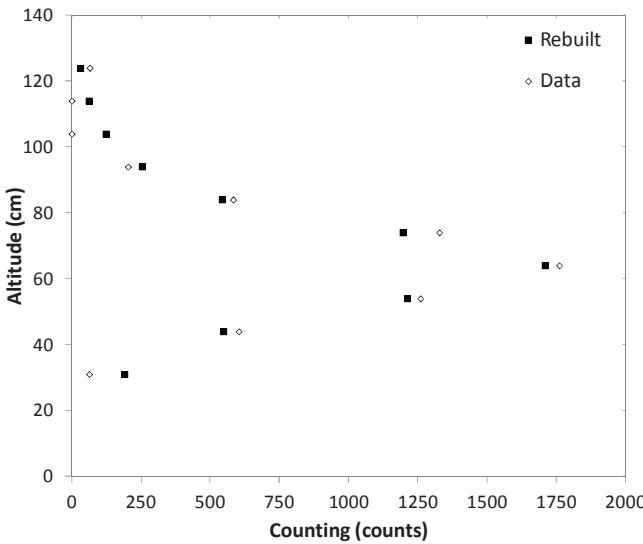


Fig. 10. Comparison between experimental data coming from the delayed neutron altitude scan and rebuilt data given by the EM algorithm.

A quantification of the global mass of uranium contained in the package has been carried out after these measurements. The actinide mass is considered to be diluted in a cylinder of 10 cm height, the dilution radius being a free parameter in order to evaluate its impact. The quantification was done, assuming a 100 %  $^{238}\text{U}$  mass. The net photofission signal was obtained, subtracting for each side the raw signal at 124 cm (assuming no  $^{238}\text{U}$  at this altitude) from the raw signal at 64 cm. Table 3 recapitulates the corresponding mass of  $^{238}\text{U}$ , according to the considered actinide repartition.

TABLE 3  
 $^{238}\text{U}$  EQUIVALENT MASS OBTAINED FROM PHOTOFISSION MEASUREMENTS,  
ACCORDING TO THE DILUTION RADIUS (HEIGHT OF THE CYLINDER IS FIXED AT  
10 CM).

Dilution radius (cm)	$^{238}\text{U}$ mass (g)
28.6	240.5
23.6	205.0
18.6	179.0
13.6	160.5
8.6	140.2
3.6	122.3
2.0	84.3

The sensitivity directly depends on the photofission rate and on the delayed neutron efficiency. The best sensitivity is obtained for a quasi-centered position which is not an intuitive result. For this position, we obtained a lower neutron efficiency but a better photofission rate in comparison with the diluted position (in this case, the actinide mass is only partially

activated, due to the reduced distance between the conversion target and the entrance of the TE 1060 block).

In order to evaluate the uncertainty linked to the knowledge of the concrete chemical composition, we simulate several variations related to the weight percentages of some key elements and we evaluate their impact on the delayed neutron efficiency (simulation of a punctual neutron source positioned at the centre of the TE 1060 block, which is the most penalizing configuration in terms of neutron detection). Table 4 recapitulates results obtained in the frame of these simulations.

TABLE 4  
IMPACT OF THE CONCRETE CHEMICAL COMPOSITION VARIATIONS ON THE DELAYED NEUTRON DETECTION EFFICIENCY (DN EFFICIENCY). ABSOLUTE VARIATIONS OF THE CHEMICAL CONTENT ARE INDICATED FOR EACH CONSIDERED ELEMENT IN THE FIRST COLUMN. INITIAL WEIGHT PERCENTAGES, CORRESPONDING TO THE REF CONFIGURATION, ARE THE FOLLOWING FOR THE STUDIED ELEMENTS: 30.1 % FOR Si, 10.0 % FOR Ca, 52.6 % FOR O AND 1.3 % FOR H.

Chemical variation	DN efficiency ( $\times 10^{-4}$ )	Ref. / DN efficiency
Ref.	1.03	1.00
Si [-10 % ; +10 %]	[0.86 ; 1.06]	[1.20 ; 0.97]
O [-10 % ; +10 %]	[1.32 ; 0.72]	[0.78 ; 1.44]
Ca [-5 % ; +5 %]	[0.88 ; 1.08]	[1.17 ; 0.96]
H [-0.5 % ; +0.5 %]	[5.47 ; 0.24]	[0.19 ; 4.38]

The impact of the chemical variations remains limited, except for the hydrogen content which has a significant impact on the quantification step. If the evaluation of this parameter remains uncertain, the use of alternative techniques (high-energy delayed gamma-ray counting, delayed gamma-ray spectrometry, passive gamma-ray spectrometry) needs to be conceived.

In order to minimize the impact of the actinide repartition on the quantification step, a photofission tomography, based on the delayed neutron detection, has been carried out [12, 13, 14]. This technique was quite well adapted to the characterization of this package, due to the concentration of the actinide mass at a given altitude. It is important to emphasize that photofission tomography has been used for the first time on a package presenting the characteristics of the TE 1060 block. Due to experimental constraints, only thirteen measurement points have been carried out, using the same neutron detection system than those described for global photofission measurements. The grid is based on 21 elemental volumes (voxels), each one being a cube of 10 cm square. For each rotation and translation position, two measurements have been carried out (one at 64 cm to obtain the raw signal, one at 124 cm to determine the active neutron background). For each position of interest, the photofission sensitivity is computed using MCNPX and the reconstruction is done using the EM

algorithm previously presented. Result of the photofission tomography is presented in Fig. 11.

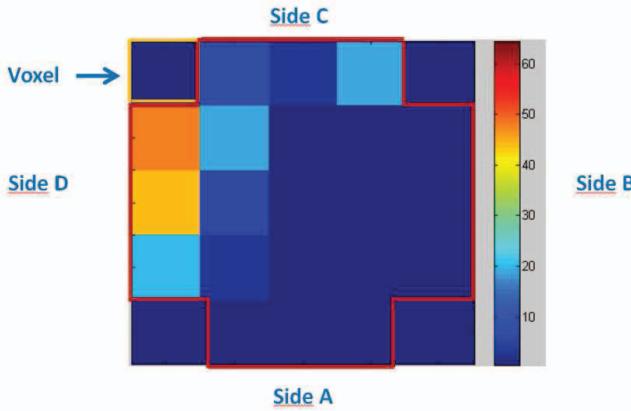


Fig. 11. Result of the photofission tomography.

Photofission tomography indicates an outlying actinide mass at the altitude of interest which is consistent with previous gamma-ray spectrometry measurements and global photofission measurements. The global  $^{238}\text{U}$  equivalent mass obtained after photofission tomography measurements is equal to 178.7 g which is the finest evaluation of the  $^{238}\text{U}$  mass contained in the package using photofission measurements.

## V. CONCLUSIONS AND FUTURE DEVELOPMENTS

In this article, we present experimental and simulated results obtained during the characterization of an old nuclear waste package identified as TE1060 block. Passive and active non-destructive methods have been used to obtain a great number of information related to the content of this block. The interest of non-destructive active methods based on a LINAC (high-energy radiography, photofission measurements) has been demonstrated in the frame of this characterization, the latter showing severe limitations related to the application of passive non-destructive techniques.

This characterization will be pursued in 2013, in order to obtain complementary information on the content of the package. First, a reevaluation of the density assumption will be done using transmission measurements with a  $^{60}\text{Co}$  source and using also the LINAC of the SAPHIR facility. Then, delayed gamma-ray spectrometry will be carried out in order to obtain an evaluation of the uranium isotopic composition.

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