

# The MADERE radio-activity Measurement Platform: Developments for a better addressing to the experimental needs.

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**Abstract**—The main goal of the Reactor Dosimetry is to provide information (reaction rates, fluence, fluence rate...) for the interpretation of experiments irradiated in critical mock-up, test reactors or power nuclear reactors. Various techniques are used, including analysis of irradiated activation or fission dosimeters whose radioactivity is measured afterwards.

The MADERE platform (Measurement Applied to Dosimetry for REactors) is a CEA facility which is dedicated to the activation dosimeters manufacturing and their activity measurement after irradiation in a nuclear reactor. The laboratory is accredited by the French Accreditation Committee for specific activity measurements of solid samples using gamma and X-rays spectrometry.

The choice of dosimeters takes into account limitations coming from the characteristics of the measurement devices. To meet experimenter's new demands, the MADERE platform set out to improve its offer by lowering the energy of measured radiations down to 10 keV, and the activity level down to the tenth of Becquerel (Bq). Doing so, the range of usable dosimeters and by the way, the energy range of the neutron spectrum is expanded.

Dosimeter, wires or foils, few millimeters large, are manufactured using ultra-pure material (Gold, Iron, Nickel,...). Some of them are encapsulated in quartz containers for integration into experimental devices.

In order to determine the activity of irradiated metal solid samples, the MADERE platform makes use of several measurement equipments:

- Three Gamma spectrometry devices equipped with semiconductor diode detectors HPGe set to work on an energy field ranging from 50 keV to 2 MeV. The range of activity covered by these devices spreads from a few tenths of Bq to a few tens of MBq. The ability to measure samples with activities of the order of one tenth of Bq may be used to implement a non-destructive determination of the isotopic composition of low-active objects.

- One X-ray spectrometry device equipped with a LEGE (Low Energy GERmanium) GeHP semiconductor detector. The settings of this equipment have been made to work in an energy range from 10 keV to 300 keV. This range allows covering almost all the X energy domain and also easily measuring low energy gamma emitters. For a long time, this device has been exclusively used for relative activity measurements of irradiated niobium using a

<sup>93</sup>Nb<sup>m</sup> reference sample (17 keV Xk emitter). This direct comparison measurement method allows getting free from the determination of different measurement parameters, since they are considered proportional between the reference and the measured samples, under the hypothesis that geometry, activity level and measurement conditions are identical. A need has emerged for measuring the <sup>103</sup>Rh<sup>m</sup> activity. It would have required the use of a reference <sup>103</sup>Rh<sup>m</sup> source which is in fact excluded due to the short half-life (56 minutes) of this radionuclide. It was therefore decided to establish the total absorption yield curve in order to perform absolute activity measurement.

- One precise micro-balance allowing the measurement of sample mass with an accuracy of few micrograms.
- One fissile measurement device equipped with 4 NaI detectors associated to an automated samples holder.

The quality of gamma spectrometry measurements mainly depends on the knowledge of the detection efficiency yield associated to the Source-Detector system geometry. Thus, the MADERE platform uses twenty different calibrated measurement geometries. In order to optimize the time spent on the experimental determination of each associated detection yield, a modeling work is underway, using the ETNA software, developed by the Henri Becquerel French National Primary Laboratory, to derive transfer efficiency and correction coincidence parameters.

The precise determination of the sample activity also requires the use of correction factors related to the effects of self absorption, sum peak and geometry. Thus, the MADERE platform has developed tools using its large experimental database which allow the accurate determination of these correction factors to be applied to the sample measurements.

Finally, in order to meet the various requirements for the monitoring of the total stored activity in the facility, but also for the traceability of the measurement results and of all the elements contributing to the elaboration of the results provided to its customers, the MADERE platform has developed computerized management tools that maximize its productivity.

**Index Terms**— CEA - Radioactivity measurement – photons – X-rays spectrometry, gamma Spectrometry - Reactor dosimetry -

## I. INTRODUCTION

The MADERE platform is dedicated to the radio-activity measurement of metal samples irradiated in test reactors, power plants and zero power reactors. These measurements are carried out on a set of samples of different types selected to

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present neutron sensitivity at different energy regions. For example, the neutron spectrum rebuilding at the studied position can be done. The table below shows the main neutron dosimeters used with their energy threshold, the reactions involved and the radioactive periods of the measured radio nuclides.

Table 1

Characteristics of the main neutron dosimeters used on the MADERE platform

Dosimeter		Reaction	Sensibility energy range (MeV)	Radioactive period
Activation	Iron	$^{54}\text{Fe}(n,p)^{54}\text{Mn}$	$E > 3 \text{ MeV}$	312.13d
	Cobalt	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$	Th + epi	5.271 y
	Copper	$^{63}\text{Cu}(n,\alpha)^{60}\text{Co}$	$E > 6 \text{ MeV}$	5.271 y
	Nickel	$^{58}\text{Ni}(n,p)^{58}\text{Co}$	$E > 2.7 \text{ MeV}$	70.82 d
	Gold	$^{197}\text{Au}(n,\gamma)^{198}\text{Au}$	Th + epi	2.6944 d
	Niobium	$^{93}\text{Nb}(n,n')^{93}\text{Nb}^m$	$E > 0.1 \text{ MeV}$	16.13 y
	Rhodium	$^{103}\text{Rh}(n,n')^{103}\text{Rh}^m$	$E > 0.7 \text{ MeV}$	56.1 m
	Indium	$^{115}\text{In}(n,\gamma)^{116}\text{In}^m$ $^{115}\text{In}(n,n')^{115}\text{In}^m$	Th + epi $E > 1.3 \text{ MeV}$	54.2 m 4.486 h
Aluminium	$^{27}\text{Al}(n,\alpha)^{24}\text{Na}$	$E > 7.3 \text{ MeV}$	14.9574 h	
Fission	$^{237}\text{Np}$	$^{237}\text{Np}(n,f)^{137}\text{Cs}$	$E > 0.6 \text{ MeV}$	30.05 y
	$^{238}\text{U}$	$^{238}\text{U}(n,f)^{137}\text{Cs}$	$E > 1.5 \text{ MeV}$	30.05 y

The main customers of the MADERE platform are:

- ✓ EDF in the framework of the monitoring program of irradiation effects on PWR vessels.
- ✓ CEA, on the one hand, for the purposes of dosimetry experiments carried out in the OSIRIS test reactor and soon in its successor the RJH reactor. On the other hand in the experiment programs carried out in the critical facilities EOLE and MASURCA.
- ✓ AREVA-TA, RES reactor operator, which will be soon in operation at the Cadarache site.

The facilities of the platform may also be used occasionally, as in the case of the re-divergence of the test reactor CABRI.

## II. DESCRIPTION OF THE MADERE PLATE-FORM

The platform MADERE is a facility classified for environmental protection. It is composed of different places each of them dedicated to a specific activity:

- ✓ Storage of raw materials and manufacture of dosimeters,
- ✓ Storage of irradiated dosimeters,
- ✓ Preparation of dosimeters before measurement,
- ✓ Weighing,
- ✓ Activity measurement using gamma and X-ray spectrometry.

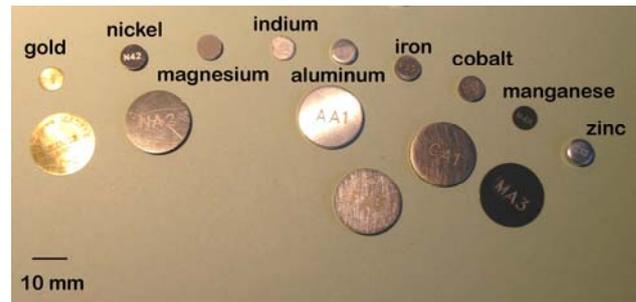
The dosimeters design is thought keeping in mind the minimization of the post irradiation activity. Regardless the benefits arising for the measurement process, that minimization allows the limitation of the doses received by the staff to extremely low levels. It also allows avoiding the use of heavy equipments (hot cells, remote manipulator etc ...) for the

handling and storage of dosimeters. Finally, it limits the induced production of radioactive waste.

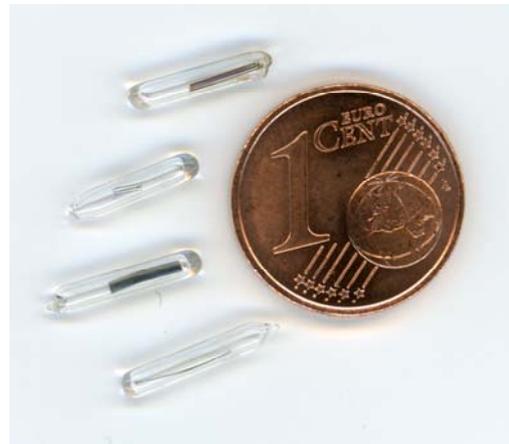
The MADERE platform is accredited by the COFRAC (French Accreditation Committee) for measuring activity of metal samples using gamma and X spectrometry.

## III. DOSIMETERS MANUFACTURING

The MADERE platform has the ability to provide its customers with all neutron dosimeters corresponding to their needs.



These dosimeters can be from 0.1 to 2 mm thick disks with diameters generally comprised between 1 and 20 mm or few millimeters long wires with diameters ranging from 0.1 to 1mm or even strips of few millimeters. They are made from very high purity materials, material certificates being available.



The wire-type dosimeters are usually enclosed in a 0.4 mm thick quartz bulb about 10 mm long and 2 mm diameter.

## IV. MEASUREMENT EQUIPMENTS

The MADERE platform is equipped with 4 high resolution gamma spectrometry devices. Three detection units are dedicated to gamma spectrometry measurement, one of them for measuring low activity samples. A fourth detection unit is used for activity measurement by X-ray spectrometry. A gamma spectrometry device with an automatically rotating holder (PASTAGA) is also available. All these devices are supplied by the CANBERRA company.

### A. The PASTAGA device

The PASTAGA device is a sample carrier designed for gamma measurement on radioactive solid samples (fissile or activation)

irradiated in low neutron flux critical mock-ups with the main objective to characterize the core.

This device consists of a carousel allowing up to 51 samples measurement per campaign. It is equipped with four NaI detectors. A 4 to 8 channels electronic permits total gamma or absolute counting under some conditions (single- energy peak and low activity).

The measurement process is controlled by a computer system specifically developed and automated in two operating modes.

- ✓ The “measure” mode allows working under the four NaI with a reference sample constantly present on the device. This mode is mainly used for the fissile samples and allows normalizing all the measurements to the reference sample. This method avoids the problem of the fission products effective period.
- ✓ The mode "program" sets any sample in front of any sensor. This operating mode is mainly used for activity measurement by gamma spectrometry on mono-energy low level activation samples.

**B. Gamma spectrometry detection units**

The three gamma spectrometry chains equipping the MADERE platform consist of hyper pure germanium detectors cooled by liquid nitrogen with resistor-type preamplifiers. The details of the detectors are presented in the table below.

Table2

Characteristics of gamma spectrometry detection units detectors

Detection unit	“Track 1”	“Carrier”	“High efficiency”
Material and type	HPGe P type coaxial	HPGe P type coaxial	Broad Energy Ge
Window	Aluminum	Aluminum	Aluminum
Relative yield at 1.33 MeV	10%	25.8 %	> 50%
FWHM at 1.33 MeV	1.69 keV	1.73 keV	1.831 keV
FWHM at 122 keV	0.782 keV	0.761 keV	0.622 keV
Energy range	50 keV to 2 MeV	50 keV to 2 MeV	50 keV to 2 MeV
Activity range	10Bq to 10 MBq	10Bq to 10 MBq	0.1Bq to 1 MBq

**Special features of the “High efficiency” system**

It is equipped with a HPGe BEGe type sensor with efficiency greater than 50%. This type of detector has simultaneously a good efficiency in the total energy field (50 keV - 2 MeV) and a limitation of the Compton effect.

The detector is placed in a circular shielding castle made of a succession of 125mm thick low noise lead, 25mm thick ultra low noise lead, 1mm thick tin and 1.6 mm thick copper.

Cooling of the detector is ensured by a cryo-cycle system providing an electric/liquid-nitrogen hybrid cooling system.

The background noise obtained on this chain is low: 1Hz.

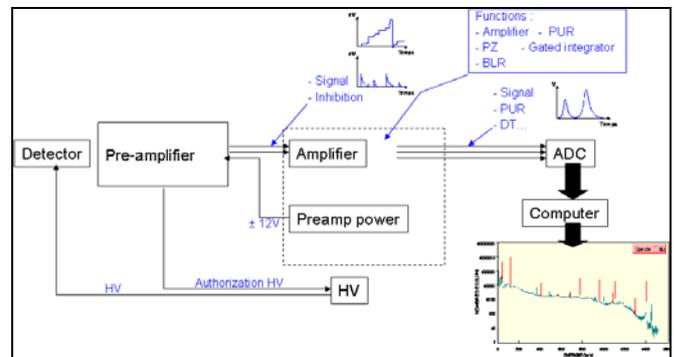
These features allow measuring easily samples with an activity of a few tenths of Becquerels.

The associated electronics is identical for all detectors and corresponds to a digital system that supports pulses directly supplied by the preamplifier. It provides the formatting and the analog/digital conversion of the signals in order to be directly usable by the multichannel analyzer (AIM) which provides the discrimination of signals in channels and the storage in a PC via an Ethernet connection.

The supply of the preamplifier with low voltage is provided by the DSP. The high bias voltage of the crystal is ensured by an electronic module similar to the ones used in the analog gamma spectrometry.

The general synopsis of the installation is shown in Figure 1.

Figure1  
Measurement detection unit synopsis



The variable settings of the DSP 2060 are numerous and allow simultaneously to optimize the shape parameters of the signal (rise time and Flat Top) and the parameters affecting the quantitative response of the system (discrimination threshold: DISC Setting; inspection time: PUR Guard; optimization of dead time correction: LT trim).

Table No. 3 shows the values of setting parameters of each of the DSP 2060 of the gamma spectrometry units.

Tableau N° 3  
Values of the main setting parameters of DSP 2060

Detection unit	“Track 1”	“Carrier”	“High efficiency”
Rise Time	5.6	5.6	2.8
Flat Top	0.8	0.8	0.9
Baseline	Auto	Auto	Auto
Conv. Gain	4096	4096	4096
PUR	ON	ON	ON
Pur Guard	2.5	2.5	2.5
LT Trim	57	43	415
Disc Thresh	Auto	Auto	Manuel
Disc Setting	1%	1%	0.6%

A the maximum counting rate encountered on the MADERE platform is not exceeding 20,000 counts per second, settings values are chosen to increase the resolution. However, they are

quite bad for dead time. For example, the maximum counting rate above leads to a dead time of about 40%.

Optimization of the dead time correction was performed using the two sources method [1] [2] which consists in disrupting a cobalt 60 source by a barium 133 source.

1) *Yield calibration*

The yield calibration is carried out every five years with a series of mono-energetic sources (<sup>241</sup>Am, <sup>109</sup>Cd, <sup>139</sup>Ce, <sup>51</sup>Cr, <sup>113</sup>Sn, <sup>85</sup>Sr, <sup>137</sup>Cs, <sup>54</sup>Mn, <sup>65</sup>Zn) to which are added sources of <sup>57</sup>Co, <sup>60</sup>Co and <sup>88</sup>Y to optimally cover the interesting energy range.

All measurement geometries are involved in this calibration, eight of them for the "Track 1" unit detection, four for the "High efficiency" unit detection and seven for "Carrier" unit detection.

Specific calibration curves are established for <sup>60</sup>Co in order to take into account the effects of sum peaks due to gamma-gamma coincidences.

The geometries of the first two systems consist of Plexiglas tubes of different heights placed on the detector. The sample to be measured is put in a cup fitted to its dimensions: this cup is placed on the top of the tube.

The "Carrier" system, as suggested by its name, consists of the combination of a measurement detection unit and a carousel allowing measuring up to 36 samples per campaign; for this system, the detector itself moves between seven positions.

2) *Controls*

In order to ensure the response stability of the measurement devices three types of periodic checks have been set;

- ✓ A monthly monitoring using a standard source made of <sup>241</sup>Am, <sup>137</sup>Cs and <sup>60</sup>Co; it allows the verification of the centroid of the <sup>241</sup>Am peak at 59 keV and of the <sup>60</sup>Co peak at 1332 keV, the energy resolution at <sup>137</sup>Cs 661 keV peak and at <sup>60</sup>Co 1332 keV peak, and finally the <sup>137</sup>Cs and <sup>60</sup>Co activity.
- ✓ A quarterly check by reiterating a sample measurement on a different measurement detection unit and by a different operator. The comparison of the two results allows the decision on the system conformity.
- ✓ At least every two years an inter laboratory test is performed, usually with the French Henri Becquerel National Laboratory (LNHB), the national reference laboratory in the field of radiation metrology.

C. *X-ray spectrometry detection unit*

The X-ray measurement detection unit is composed of a hyper pure germanium diode (HPGe), planar LEGe (Low Energy Germanium) type.

Table 4

Characteristics of the X-ray spectrometry system detector

Chain	X detection unit
Material and type	GeHP Planar LeGe
Window	Beryllium (0.5 mm)
Dimensions	Diam = 43.7mm th. = 15mm
FWHM à 5.9 keV	0.302 keV
FWHM à 122 keV	0.528 keV
Energy range	6 keV to 300 keV
Activity range	10 Bq to 1 MBq

The electronics associated to this detector are the same as these of gamma spectrometry systems.

Table No. 5 shows the values of setting parameters of the CSP 2060, part of the X-ray spectrometry system.

Table 5

Values of the main setting parameters of X ray system DSP 2060

Detection unit	X detection unit
Rise Time	6.4
Flat Top	0.7
Baseline	Auto
Conv. Gain	4096
PUR	ON
Pur Guard	2.5
LT Trim	300
Disc Thresh	Auto
Disc Setting	1%

Given the settings retained during the optimization phase of qualitative performances, on the one hand, and the activities measured on the platform MADERE on the other hand, we have limited the quantitative characterization of the system functioning in an operating range not exceeding 14,000 counts per second which corresponds to a dead time of about 30%.

Optimization of the dead time correction was performed using the two sources method [1] [2] which consisted for this measurement detection unit in disrupting a <sup>51</sup>Cr source by a <sup>109</sup>Cd source.

1) *Yield calibration*

This detection unit was used exclusively for comparative measurement between niobium samples irradiated in a reactor and a <sup>93</sup>Nb<sup>m</sup> standard (X-ray emitter at about 17 keV). This direct comparison measurement method allows to take into account peaks shape parameters and possible loss of counts, since they are considered proportional between the reference and the sample, provided that their geometry and their measurement conditions are identical.

For this method, a <sup>93</sup>Nb<sup>m</sup> source regularly calibrated by the LNHB and three so-called "work standards" whose geometry is identical to that of samples usually measured by the laboratory and to that of the standard reference are used.

The calibration of these "work standards" is done by the MADERE platform using the standard reference at least every five years.

The need of <sup>103</sup>Rh<sup>m</sup> activity measurement [3] led us to achieve absolute calibration of the measurement device with <sup>241</sup>Am,

$^{109}\text{Cd}$ ,  $^{57}\text{Co}$ ,  $^{93}\text{Nb}^m$ ,  $^{137}\text{Cs}$  et  $^{133}\text{Ba}$  sources whose X-ray emission has been characterized by LNHB.

The samples positioning is realized using a removable device that can meet all needs:

- ✓ Ensure good repeatability of the source/detector positioning to minimize the positioning errors,
- ✓ Allow positioning calibration sources and samples at the same distance from the detector,
- ✓ Allow the positioning of collimators at a distance such as the measurement solid angle is smaller than or equal to 0.18 Sr (calibration condition).
- ✓ The holder must be as light as possible and be build with a light material to minimize the diffused radiations,
- ✓ Do not interfere with the  $^{93}\text{Nb}^m$  relative measurement process.

The device is made of Plexiglas (methyl polymetacrylate,  $(\text{C}_5\text{H}_8\text{O}_2)_n$ ). It is equipped with three identical uprights with rabbets every centimeter in order to position the collimators and the cups containing sources or samples. The three uprights are maintained by two rings.



Sample positioning device

#### 2) Controls

As for the gamma spectrometry detection units, the stability of the system response is checked periodically by:

- ✓ a monthly check which allows, using a standard source of  $^{133}\text{Ba}$ , to verify the value of the centroids, the resolution and the areas for the 35 and 302 keV peaks.
- ✓ at least every two years an inter laboratory test is performed, usually with the LNHB.

As we have only one X-ray spectrometry device, no reiterated measurements are carried out.

#### D. Micro balance

The results provided by the MADERE platform in the analysis reports are **mass activities** (Bq/mg). Thus, the determination of the dosimeter mass is an important element in the process.

To achieve these mass measurements, the laboratory is equipped with a precision micro balance METTLER MT5 ( $1\mu\text{g}$  to  $5000\mu\text{g}$ ). The design of this balance, in particular the fact that all electronics elements are separated and do not warm the weighing room, avoids the effect of samples acclimation (the temperature change of the weighing chamber is only  $0.1\text{ }^\circ\text{C}$ ). Moreover, the cylindrical design of the weighing chamber windscreen reduces the effect of thermal radiation such as those coming from the operator. Finally the micro balance is equipped with a special device for automatic calibration when the integrated sensors detect a temperature change.

The balance is connected by a RS232 connection to a computer allowing the integration of the masses in a database that includes all the information on the measured samples at the MADERE platform.



Balance METTLER MT05

#### 1) Controls

The platform owns standard masses (with calibration certificate). They are verified versus primary standards every three years by a calibration laboratory accredited by an external company, such as the mass metrology laboratory of the French National Testing Laboratory accredited by the French "COFRAC/Etalonnage".

Three types of checks are carried out periodically for the balance:

- ✓ Before each set of weighing, two standard masses are weighed and the obtained values are checked to be within the tolerance. The standards are chosen such as the samples masses are situated between the standard ones.
- ✓ Quarterly, controls on the rightness and fidelity allowing determining the equipment compliance.
- ✓ Annually a control by a maintenance technician of the METTLER Company.

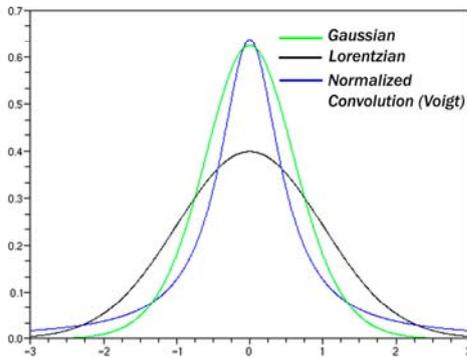
All records related to these controls are stored.

#### V. DATA PROCESS

The MADERE platform uses the software "GENIE 2000" [4] of the CANBERRA Company as acquisition software for all gamma and X-ray spectrometry measurements but also for gamma spectra process.

However, X-ray and gamma ray have different origins resulting in different peak shape. The peaks due to gamma emission are adequately represented by a mathematical Gaussian function due to the detection process. X-ray peaks must be described by

a Voigt function, resulting of the convolution of a Lorentzian shape by a Gaussian shape due to the detection process.



#### Mathematical function shape differences between a Gaussian and a Voigt type

The “GENIE 2000” software can only process the Gaussian shaped peaks (fitted to gamma photons). It can be used for <sup>93</sup>Nb<sup>m</sup> measurement by direct comparison (relative method). In no case it should be used for absolute processing of X photons peaks and it is necessary to use a specific application to process Voigt shape.

We use the specific software COLEGRAM developed by the Henri Becquerel National Laboratory (LNHB) [5]. It allows adjusting different functions (about twenty kinds of shapes - Gaussian with or without tail, exponential, polynomial, Voigt...) to experimental points of a spectrum. The adjustment is done according to least squares criterion with the ability to freeze some parameters characterizing the studied function (energy, amplitude, peak width at half height, natural width for X-ray peaks ...). This feature allows a large possibilities in the peaks processing, but requires a very good knowledge of the spectrum to be studied and the measurement environment.

#### A. Determination of the samples activity from measurements by gamma spectrometry

For the <sup>j</sup>th peak, the activity A<sub>j</sub> of the radionuclide, expressed in Becquerels, is calculated at the origin date according to the relation:

$$A_j = \frac{N(E_j)}{t \cdot I_\gamma(E_j) \cdot \varepsilon(E_j)} \cdot C_m \cdot C_a \cdot C_{Plex2mm} \cdot C_{geo} \cdot C_{abs}(E_j) \cdot C_{som}(E_j)$$

where:

- N(E<sub>j</sub>) = net area under the total absorption peak at energy E<sub>j</sub> (counts),
- t: counting time (s),
- I<sub>γ</sub>(E<sub>j</sub>): emission probability of the photon with energy E<sub>j</sub> of the studied radionuclide; I<sub>γ</sub>(E<sub>j</sub>) values come from [6].
- ε(E<sub>j</sub>): total absorption yield for the photon with energy E<sub>j</sub> for the measurement geometry,

- C<sub>m</sub>: correction factor for the decay of the radionuclide

$$\text{during the measurement: } C_m = \frac{\lambda \cdot t}{1 - e^{-\lambda \cdot t}}$$

with λ: radionuclide decay constant, coming from [6].

Nota: for most of the measured radionuclides at the MADERE platform, C<sub>m</sub> is equal to 1.

- C<sub>a</sub>: correction factor for the decay of the radionuclide coming from the cooling time (a: time between the origin date and the beginning of the measurement):

$$C_a = e^{\lambda \cdot a}$$

C<sub>geo</sub>: correction factor for the shape difference between the sample and the standards used for the yield curve elaboration,

- C<sub>abs</sub>(E<sub>j</sub>): correction factor for self-absorption of photons with energy E<sub>j</sub> in the sample,
- C<sub>som</sub>: correction factor for the sum effects due to photons emitted in cascades.
- C<sub>Plex 2mm</sub>: correction factor for the difference between the cups used for standards and for samples (for the “Carrier” detection unit only).

When several peaks are used, the expression of the final result is a weighted average of the individual activity A<sub>j</sub> obtained for the different total absorption peaks.

Among the terms used for calculating the activity, some are taken into account directly by the processing software (GENIE 2000) using the parameters provided for the acquisition: N(E<sub>j</sub>), ε(E<sub>j</sub>), C<sub>m</sub>, C<sub>a</sub>.

The others, C<sub>geo</sub>, C<sub>som</sub>, C<sub>Plex 2mm</sub> and C<sub>abs</sub>, are determined during the analysis report elaboration.

#### 1) Determination of correction factors C<sub>geo</sub>, C<sub>abs</sub>, C<sub>som</sub>, C<sub>Plex 2mm</sub>.

##### a) Geometry correction factor C<sub>geo</sub>

The efficiency curves of a gamma spectrometry detection unit are elaborated with standard sources considered having no thickness; as it is not the case for the samples measured by the MADERE platform, this geometrical difference must be taken into account while determining the samples activity.

The difference being of a few tenths of mm, the geometry correction factor is calculated by considering either the half-thickness for discs or the radius for wires. The geometry correction factor is determined using a single energy standard source (for example <sup>137</sup>Cs) among the used standard sources set. The determination of this correction is based on the dissymmetry of the standard sources holder.

The correction factor for the geometry correction is determined by the following formula:

$$C_{geo} = 1 + \left( 1 - \frac{A_2}{A_1} \right) \cdot \frac{0.1}{\Delta}$$

A<sub>1</sub>: <sup>137</sup>Cs source activity obtained with the serial number oriented downwards.

A<sub>2</sub>: <sup>137</sup>Cs source activity obtained with the serial number oriented upwards.

Δ: source height difference (in mm) between upwards and downwards countings.

b) *Self-absorption correction factor C<sub>abs</sub>*

Self-absorption stands for the attenuation inside the sample material. The standard sources used for efficiency calibration having no thickness, this effect is considered as negligible.

The correction factor, for samples, is:

$$C_{abs} = \frac{1}{1 - a_s}, \text{ where } a_s: \text{ self-absorption fraction.}$$

For the samples shape commonly used at the MADERE platform, the a<sub>s</sub> factor is expressed by the following formulas :

- Disks:

For disks whose thickness (t) is small compared to the sample-detector distance and to the sample diameter:

$$a_s = 1 - \frac{1 - e^{-\mu \cdot t}}{\mu \cdot t}$$

- Wire with radius r :

$$a_s = \frac{8}{3 \cdot \pi} \cdot \mu \cdot r - \frac{1}{2} \cdot \mu^2 \cdot r^2 \text{ for } \mu \cdot r \ll 1$$

Attenuation coefficients (μ) come from NIST XCOM tables.

c) *Gamma-gamma coincidence correction factor C<sub>som</sub>*

This phenomenon results from the simultaneous detection of two photons emitted in a cascade. Its occurrence is all the more frequent that the counting geometry is close (large solid angle) and that the disintegration scheme has several cascades.

The correction is done either analytically or using the ETNA software [7] developed by the Henri Becquerel National Laboratory (LNHB).

d) *C<sub>plex 2mm</sub> correction factor*

This correction allows taking into account the difference between the cups used during calibration and those fitted for samples measurement, only for the “carrier” detection unit.

The correction factor is obtained by an attenuation calculation through a 2 mm thickness of the cups materials (which are identical) as a function of the considered radiations energy.

B. *Samples activity determination from X-ray spectrometry measurements.*

<sup>93</sup>Nb<sup>m</sup>

X-ray spectrometry measurement results are sample X-ray emission rate. The sample and a “work standard”

with the emission rate is well known, are both measured in the same conditions.

For the i<sup>th</sup> sample, the <sup>93</sup>Nb<sup>m</sup> activity a<sub>i</sub> (Bq/mg) is calculated at the origin date by the formula:

$$a_i = \frac{1}{m_i} \cdot \frac{N_i / t_i}{N_{ref} / t_{ref}} \cdot C_{ep} \cdot C_{fluo} \cdot A_{ref}$$

where : N<sub>i</sub>: sample counts under the 16,5 keV peak,

N<sub>ref</sub>: standard counts under the 16,5 keV peak,

m<sub>i</sub>: sample weight (mg),

C<sub>ep</sub>: correction factor for the thickness difference between the sample and the standard,

C<sub>fluo</sub>: fluorescence correction factor,

t<sub>i</sub>: sample acquisition time (s),

t<sub>ref</sub>: standard acquisition time (s).

1) *C<sub>ep</sub> and C<sub>fluo</sub> correction factors determination*

a) *Correction factor for the thickness difference between the sample and the standard C<sub>ep</sub>*

This correction allows taking into account thickness differences that may exist between the sample and the standard. The determination of mass absorption coefficients for the evaluation of this correction has been achieved experimentally using the monochromatic X-ray source SOLEX (Source Of Low Energy X-rays). This facility, tunable in a wide range of energy, has been developed and is operated by the Henri Becquerel National Laboratory (LNHB) [8].

b) *C<sub>fluo</sub> fluorescence corrective factor*

Different impurities inside the sample may perturb <sup>93</sup>Nb<sup>m</sup> measurement. A gamma spectrometry measurement is performed for each dosimeter to determine the correction factors to be taken into account. The origin date of the activity is the start time of the sample measurement. For this correction the interesting radio nuclides are:

Nuclide	T <sub>1/2</sub>	γ Peak(s) (keV)
<sup>94</sup> Nb	20 000 y	702,62 / 871,09
<sup>95</sup> Nb	34.99 d	765,8
<sup>92m</sup> Nb	10,15 d	934,44
<sup>182</sup> Ta	114,43 d	1121,30 / 1221,41

**Activity determination using absolute method**

The correction factors to be used with the activity absolute measurement method are the same as these for gamma spectrometry.

VI. UNCERTAINTIES ASSOCIATED TO MASS ACTIVITY VALUE

The various terms used in the activity determination are affected by uncertainties that must be taken into account in the

expression of the final result. It is necessary to get a vision as complete as possible on uncertainty causes.

In the case of non-correlated data, the composed uncertainty is expressed as following for the function  $y = f(x)$ :

$$u_c^2(y) = \sum_{i=1}^n \left( \frac{\partial f}{\partial x_i} \right)^2 u^2(x_i)$$

For the uncertainty associated to a total absorption peak activity, the choice has been made to use the value calculated by the GENIE 2000 software

#### A. Calculated activity uncertainty

For the determination of a radionuclide activity, the A-type uncertainties relate to the total absorption peak area, and B-type to the following parameters:

- total absorption efficiency at the considered peak energy,
- gamma emission probability,
- decay correction for sample cooling,
- correction for radionuclide decay during the measuring time.

#### B. Correction factors uncertainties

These B-type uncertainty components determination are based on other means (experience, manufacturer specifications, certificates ...). They are characterized by evaluated standard deviations  $u_i$ . When the values are expanded uncertainties,  $U_i$  is obtained by dividing the indicated value by the enlargement factor, when specified.

For the determination of a radionuclide activity, B-type uncertainties concern the following terms:

- self-absorption correction factor,
- counting geometry correction factor,
- sum peak correction factor,
- reproducibility of measurement distances (counting geometry),
- sample centering,
- reproducibility of a reference standard measurement,
- special case when total absorption peak area is lower than 15 000 counts.

#### C. Weighting uncertainty

It takes into account the following elements:

- Uncertainty associated to the balance linearity error,
- Uncertainty associated to the resolution,
- Uncertainty associated to the calibration error,
- Uncertainty associated with the elementary uncertainty of the closest reference standard mass to the samples masses to be weighed,
- Uncertainty associated to the operator,
- Uncertainty associated to the drift during the weighing of the calibration mass.

The following table shows the order of magnitude of the elementary uncertainty, function of the mass value.

Table 6 : Order of magnitude of the elementary uncertainty, function of the mass value

Mass value	1 mg	10 mg	50 mg	100 mg	500 mg
order of magnitude of the elementary uncertainty	0,6 %	0,06 %	0,012 %	0,006 %	0,002 %

## VII. CONCLUSIONS

The MADERE platform, fitted out with different devices, is a powerful tool for reactor dosimetry. The ranges covered by the platform in terms of activity (0.1 Bq to 10 MBq) and energy (10 keV to 2 MeV) can meet all the dosimetry requirements as well for critical mock-ups as for test reactors or power plants.

Improvements planned in the near future deal, in the one hand, with the development of new types of dosimeters with dimensions compatible with easy handling and allowing low activities for high neutron fluences. On the other hand, in order to optimize the work concerning the yield calibration, it is intended to model some geometries with a Monte Carlo code.

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