

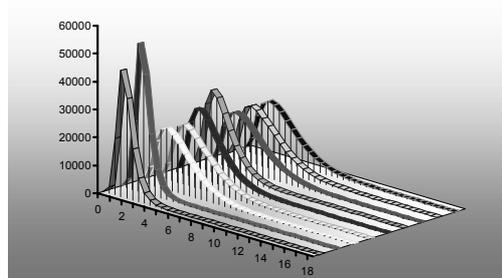
## TRITIUM EVALUATION IN GRAPHITE RADIOACTIVE WASTE BY MICROWAVE ACID DIGESTION

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A detailed analysis technique for tritium recovery from contaminated graphite (similar to irradiated reactor graphite) was established using microwave acid digestion followed by liquid scintillation counting (LSC). Two types of liquid scintillation cocktails were tested. For an added tritium radioactivity of  $8.874 \cdot 10^4 \text{ Bq} \cdot \text{L}^{-1}$  ( $2.218 \cdot 10^3 \text{ Bq}$  per graphite sample), equivalent to tritium radioactivity in graphite wastes collected from thermal column of Roumanian TRIGA (Training, Research, Isotopes, General Atomics) 14 MW research reactor, the recovery percent range of tritium was around 97 %.



### INTRODUCTION

Tritium in various kinds of samples can be detected through liquid scintillation counting (LSC), which is an analytical technique defined by the incorporation of the radioactive sample into a homogenous distribution with the appropriate liquid scintillation cocktail.

The graphite waste must be solubilized prior to dissolving into the liquid scintillation cocktail. The graphite waste is considered as a difficult-to-dissolution sample, therefore, strong acid mixture, high pressure and temperature conditions are necessary for a complete dissolution. Different decomposition methods of the graphite samples have been reported in the literature such as: combustion in presence of a catalyst in a tubular oven,<sup>1-5</sup> combustion using a sample oxidizer equipment,<sup>6</sup> acid digestion using a mixture of inorganic acids.<sup>6-7</sup> To assure homogeneous samples, all these decomposition methods use graphite in fine powder form.<sup>1-7</sup>

It is apparently that no application of microwave acid digestion for decomposition of graphite and tritium recovery evaluation has been reported. The feasibility of analysis techniques that involve microwave digestion system and LSC on graphite samples is investigated in this paper.

The microwave digestion process was employed in order to completely decomposition of the graphite matrix and to completely transfer the radionuclide into solution so that LSC technique can be used. The digestion vessels must be made of a Teflon material or quartz glass in order to ensure both chemical as well as temperature stability. Also, absorption and adsorption effects are significantly reduced. The samples in microwave digestion equipment are heated directly by the absorption of microwave radiation, applying appropriate parameters (temperature, pressure, time, slope, power level). The microwave effect or advantage of microwave heating consists in simultaneous heating 8 to 12 numbers of samples. The temperature progression of each sample must

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be continuously recorded and the microwave power must be regulated accordingly because the rapid heating is accompanied by an equally rapid pressure increase and spontaneously induced exothermic reactions. The pressure measurement can be employed to influence power regulation and thus an optimal process control can be achieved from a safety aspect.<sup>8</sup>

This paper focused on development of a method for the evaluation of  $^3\text{H}$  radioactivity in contaminated inactive graphite similar to the irradiated graphite from the thermal column of TRIGA reactor. The TRIGA reactor is Training, Research, Isotopes, General Atomics 14 MW research reactor located at the Institute for Nuclear Research Pitești.

The thermal column of TRIGA reactor is a 1716x1144x710 mm graphite block and consists of 96 rectangular graphite cells (144x144x770 mm, 12 rows, 8 bricks per row) in aluminum cladding, placed in the reactor pool.<sup>9,10</sup>

A flat drill is suggested to be used to collect samples in the form of fine powder. To avoid contamination, the surface material is not recommended to be collected. Thus, representative homogenous samples for appropriate location of the cell can be obtained and no tritium is lost from these samples. It is essential to collect the dust using suitable suction and filter systems.<sup>1-7</sup>

Main contribution to accumulation of tritium in irradiated graphite is given by activation of  $^6\text{Li}$  impurities in graphite matrix. There is a high probability that the generated tritium will remain in the graphite matrix because mean free path of tritium in the  $^6\text{Li}(n,\alpha)^3\text{H}$  reaction is smaller than size of graphite grains and larger than size of pores.<sup>6,11</sup>

Furthermore, it was investigated the stability of  $^3\text{H}$  in the fine powder and the results showed no significant change of tritium content, therefore  $^3\text{H}$  in graphite is quite stable.<sup>6</sup>

The  $^3\text{H}$  presence in the irradiated graphite depends on its microstructure and impurities distribution. Microstructure analysis of the bulk graphite revealed a heterogeneous structure in which the micro-plates are mixed with micro-bares. The magma-like aspect is specific for the fabrication process. The impurities seem to be concentrated preferentially in the pores.<sup>12</sup> Tritium does not appear to be integrated into the graphite crystal lattice but only adsorbed on the surface on

graphite crystallites and of pores, not involving chemical bounds.<sup>13-15</sup>

Tritium is distributed non-uniformly in the graphite cells, depending upon their position to the reactor core and irradiation history. Therefore, it is necessary to investigate axial and radial distributions of tritium in these cells.<sup>12</sup> Detailed information on the sampling locations has been presented in a previous paper.<sup>9</sup>

Regardless of creation, adsorption and retention of  $^3\text{H}$  on/into the graphite matrix, it is possible to remove all tritium content through the application of destructive techniques which compromise the structural integrity of the graphite matrix.<sup>1-7, 14, 16</sup>

## EXPERIMENTAL

### 1. Equipments and Chemicals

A Speedwave<sup>®</sup> four microwave acid digestion system was used to decompose the graphite samples. A 3110 Tri-Carb<sup>®</sup> PerkinElmer liquid scintillation analyzer, with minimum detection efficiency for  $^3\text{H}$  of 60 %, was used to measure tritium activity. Ultima Gold<sup>®</sup> XR and Ultima Gold<sup>®</sup> AB scintillation cocktails were tested for tritium evaluation in solution obtained after graphite mineralization.  $^3\text{H}$  standard solution (provided by "Horia Hulubei" National Institute of Research and Development for Nuclear Physics and Engineering, Bucharest), where  $^3\text{H}$  exists as HTO form, was used. Other chemicals used were analytical grade reagents.

### 2. Analysis method

Tritium may exist in the irradiated graphite from thermal column of TRIGA reactor as HT and HTO forms.<sup>6, 12, 17</sup> The HT form is easily converted to HTO form, by exchange (in presence of water vapors) and oxidation reactions.<sup>18,19</sup> Furthermore, the release of tritium from graphite is caused by graphite decomposition methods that lead to release of tritium as HTO form.<sup>1-7</sup>

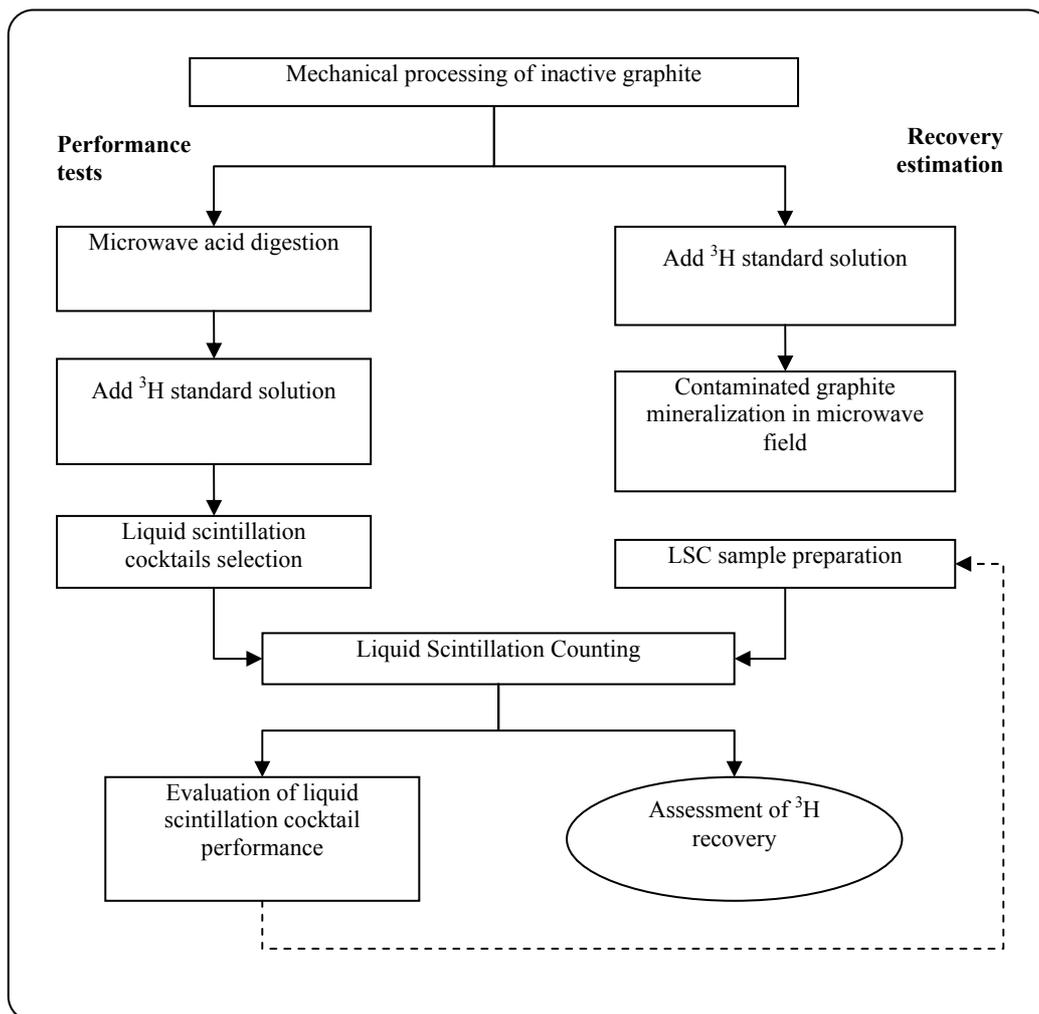
Therefore, the procedure developed in this paper uses inactive graphite and  $^3\text{H}$  standard solution, in which  $^3\text{H}$  exists as HTO form. The non-radioactive graphite is the same type as that used in the thermal column of TRIGA reactor.

The flow diagram of the proposed procedure is shown in Fig. 1.

The inactive graphite samples were mechanically processed (flat drilled) to fine powder (with diameter of particle less than 0.2 mm).

Performance tests were run to choose the scintillation cocktail.  $^3\text{H}$  standard solution was added to the digested inactive graphite samples to reveal the performance of liquid scintillation cocktails regarding their compatibility with solubilized graphite.

After the scintillation cocktail was selected, recovery estimation tests were performed to evaluate the  $^3\text{H}$  activity recover in the contaminated graphite samples. In this case,  $^3\text{H}$  standard solution was added to inactive graphite prior the mineralization process.

Fig. 1 – Flow diagram of  $^3\text{H}$  recovery evaluation procedure.

### 3. Performance tests

#### 3.1. Microwave acid digestion

Three inactive graphite samples of 0.05 g were weighted and placed into three 100 mL digestion vessels to test the repeatability of the digestion process. A 25 mL mixture of concentrated acids,  $\text{HNO}_3$ :  $\text{H}_2\text{SO}_4$  = 1:4, was used to digest each of the three samples. A rupture disc placed in the vessel cap limits the interior pressure up to the maximum permissible programmed value for each vessel. Acid vapors that may escape when the rupture disc activates are guided to a collection system integrated in the rotor, and further the vapors are vented through the PFA tube connected to the swiveling lid. In these experiments no leakage of the vapors

was observed during and after the digestion process. PFA is a perfluorated plastic (PTFE) with perfluoroalkoxy side chain (4...10% by weight).

The operational parameters of the microwave equipment applied to graphite mineralization are presented in Table 1.

The digestion program was performed in 5 steps and it was completed in 80 minutes.

For each step of the digestion program the temperature control module of the equipment powers the microwave energy into the digestion vessels. The temperature set point is achieved at the end of the programmed ramp time and kept constant at that level for the preset amount of time.

Table 1

Operational parameters of microwave acid digestion procedure

Step no.	Set point temperature T [°C]	Maximum allowed pressure P [bar]	Slope (ramp time of the program step) [min]	Time for which T is kept constant [min]	Electrical power [%]
1	180	70	10	5	70
2	220	70	10	8	75
3	250	80	5	30	85
4	50	60	1	10	0
5	50	0	1	0	0

After the microwave acid digestion was completed, the vessels were kept to room temperature to avoid gas generation. The vessels were opened and clear solutions were observed in all three of them. One solution from a digestion vessel (arbitrarily chosen) was used to investigate the liquid scintillation cocktail performance, according to the sequential procedure presented in Fig. 1.

### 3.2. Liquid scintillation cocktails selection and counting

Selection of an appropriate scintillation cocktail is very important for the accurate results in LSC technique.

Two types of liquid scintillation cocktails (Ultima Gold<sup>®</sup> XR and Ultima Gold<sup>®</sup> AB) were tested to evaluate their compatibility with chemical composition of solution resulted by microwave acid digestion. The liquid scintillation cocktails were mixed both with solubilized graphite samples and with solutions prepared by dilution from these samples, applying different dilution factors (10, 15 and 20) and different loading factors (0.5 mL and 1mL).

Eight sample sets were prepared for LSC, each of them consisting in three aliquots, resulting 24 samples with codes **ir**, where **i** represents the code of the set, **i** = {1, 2, ...8}, and **r** represents the code of the aliquot, **r** = {a, b, c}; all the 24 samples were labeled with <sup>3</sup>H standard solution. A sample set of <sup>3</sup>H standard solution consisting in three aliquots marked as E1, E2 and E3 were prepared for LSC, too.

In Table 2 are presented the reagents volumes employed in order to obtain compatible samples with LSC technique.

All the 27 samples were counted by LSC analyzer. The sample measuring statistical precision, 2s%, of 0.50 % was programmed as ending condition of the counting.

### 4. Recovery estimation

An inactive graphite sample was contaminated with tritium using <sup>3</sup>H standard solution. From this graphite waste, two aliquots were sampled and processed by microwave acid digestion. The same operational parameters of the microwave acid digestion process as in evaluation of liquid scintillation cocktail performances were applied in this phase of the experiment (see Table 1).

From the solutions resulted after mineralization of graphite waste, two sample sets (marked with G1, G2, and each of them consisting in three aliquots, marked as .1, .2 and .3) were prepared as follow: solutions were diluted with distilled water (dilution factor of 20), 0.5 mL volumes from these diluted solutions were pipetted into 20 mL LSC vials and Ultima Gold<sup>™</sup> XR cocktail was added from dispenser and mixed by shaking the vials.

All the six vials were counted in the LSC analyzer, with a sample measuring statistical precision, 2s%, of 0.50 % as an ending counting condition.

Table 2

Components and labeling of samples analyzed by LSC

Sample code		Dilution Factor	Loading Factor		<sup>3</sup> H standard [mL]	Ultima Gold <sup>™</sup> XR [mL]	Ultima Gold <sup>™</sup> AB [mL]
Set	Aliquot		Mineralized solution [mL]	Distilled water [mL]			
E	1	No dilution	NA <sup>a</sup>	NA	0.075	18.5	NA
	2		NA	NA	0.075	18.5	NA
	3		NA	NA	0.075	18.5	NA
1	a	No dilution	0.075	NA	0.075	18.5	
	b		0.075	NA	0.075	18.5	
	c		0.075	NA	0.075	18.5	
2	a	No dilution	0.075	NA	0.075		18.5
	b		0.075	NA	0.075		18.5
	c		0.075	NA	0.075		18.5
3	a	10	0.5		0.075	18.5	CS <sup>b</sup>
	b		0.5		0.075	18.5	CS
	c		0.5		0.075	18.5	CS
4	a	15	0.5		0.075	18.5	CS
	b		0.5		0.075	18.5	CS
	c		0.5		0.075	18.5	CS
5	a	15	1		0.075	18.5	CS
	b		1		0.075	18.5	CS
	c		1		0.075	18.5	CS
6	a	20	0.5		0.075		18.5
	b		0.5		0.075		18.5
	c		0.5		0.075		18.5
7	a	20	0.5		0.075	18.5	
	b		0.5		0.075	18.5	
	c		0.5		0.075	18.5	
8	a	20	1		0.075	18.5	CS
	b		1		0.075	18.5	CS
	c		1		0.075	18.5	CS

<sup>a</sup> NA – non-applicable sample

<sup>b</sup> CS – colored sample

## RESULTS AND DISCUSSION

All reported uncertainties in the paper,  $\pm U$ , are expanded uncertainties as defined in VIM,<sup>20</sup> calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

### 1. Evaluation of liquid scintillation cocktail performance

The arithmetic means of counting efficiency (as a function of quenching) and relative deviations of the  $^3\text{H}$  activity in graphite samples compared to arithmetic mean of  $^3\text{H}$  activity in standard solution were calculated (Table 3).

The counting efficiency is defined as the ratio of the number of observed counts per minute (CPM) to the number of disintegrations per minute (DPM) occurring in the sample.

The relative deviation is defined as the ratio between the absolute difference of standard activity and sample activity, and the standard activity.

The combined uncertainties of the sample activity and standard activity were calculated as the ratio between the standard uncertainty of the counting  $2s\%$  and the square of the number of replicas, as type A evaluation of uncertainty, for repeated observations.<sup>21</sup>

The combined uncertainties for the counting efficiency and relative deviation were calculated applying the rules of expressions models involving a sum or difference, a product or quotient quantities and the combination of them.<sup>22</sup>

#### 1.1. Comparative test for non-diluted samples

Very low counting efficiencies because of quench, and a relative deviation greater than 45 % were obtained for non-diluted sample set no. 1 (prepared with Ultima Gold™ XR), and no. 2 (prepared with Ultima Gold™ AB).

Fig. 2 shows quenched beta-spectra of  $^3\text{H}$  of the 1 and 2 samples compared to the sample E.

Due to quench, the energies spectra detected from  $^3\text{H}$  are shifted toward very low energies (1.94 keV for sample 1 and 1.90 keV for sample 2 compared to 3.70 keV value for tritium in the sample E).

#### 1.2. Comparative test for diluted samples

A dilution factor comparison test was performed. A decrease of relative deviation with increase of dilution factor, for 0.5 mL sample volumes and Ultima Gold™ XR as liquid scintillation cocktail, was observed. The dilution factors used were 10 for sample code no. 3, 15 for sample 4, and 20 for sample 7.

Fig. 3 illustrates beta-spectra of  $^3\text{H}$  of the 3, 4, 7 samples relative to the sample E.

Table 3

Counting efficiency and relative percentage deviation of the  $^3\text{H}$  activity in radioactive samples

Sample code	Counting efficiency [%]	Sample activity <sup>a</sup> [dpm]	Relative deviation [%]
E	44.30 ± 0.18	96727.34 ± 279.23 <sup>b</sup>	-
1	7.16 ± 0.03	52866.33 ± 152.67	45.34 ± 0.16
2	5.08 ± 0.02	44774.67 ± 130.07	53.71 ± 0.13
3	40.41 ± 0.16	88100.67 ± 254.33	8.92 ± 0.26
4	41.52 ± 0.17	90310.33 ± 260.73	6.63 ± 0.27
5	32.46 ± 0.13	83747.33 ± 241.78	13.42 ± 0.25
6	29.01 ± 0.19	87082.33 ± 251.49	9.97 ± 0.26
7	44.50 ± 0.18	96578.67 ± 278.81	0.15 ± 0.29
8	44.40 ± 0.18	95326.67 ± 275.19	1.45 ± 0.28

<sup>a</sup>  $^3\text{H}$  activity average values in each of the eight sample sets presented in Table 2

<sup>b</sup> Average value of the  $^3\text{H}$  activity in the standard solution set

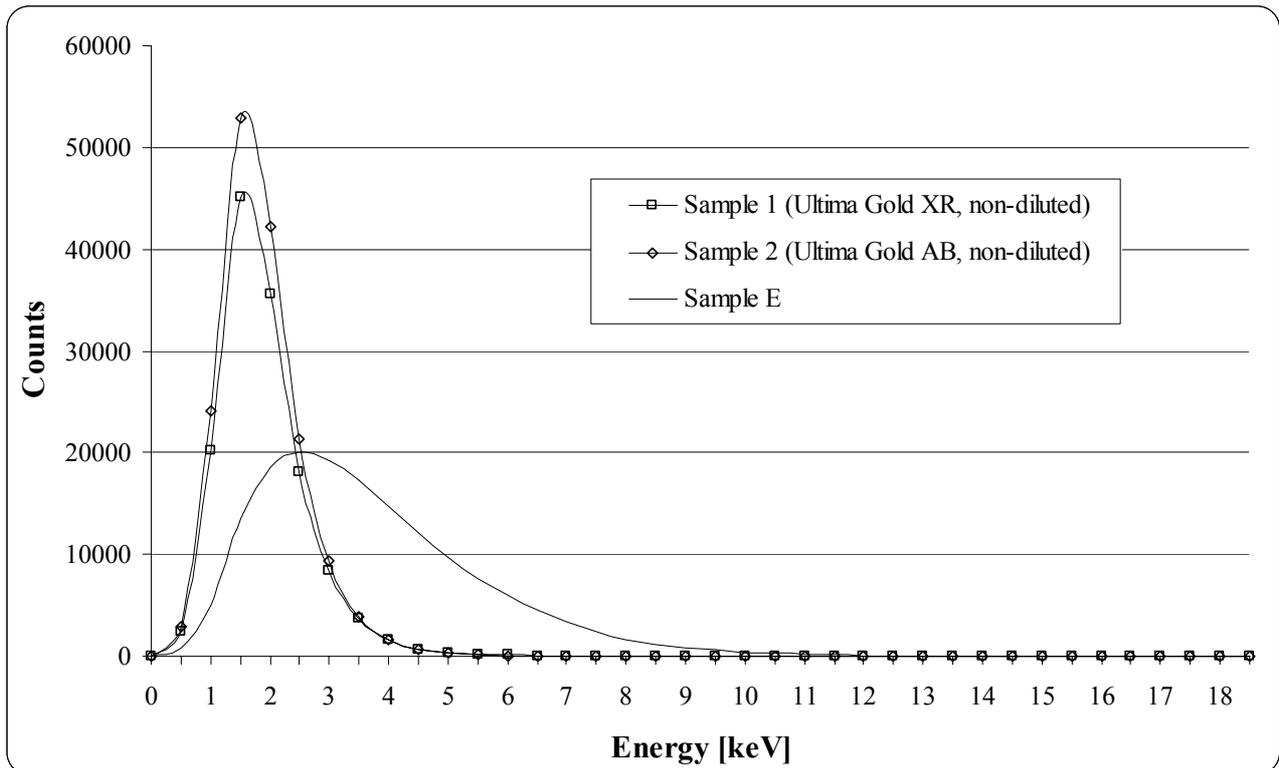


Fig. 2 – Quenched beta energy spectra of  $^3\text{H}$  in non-diluted 1 and 2 samples compared to E sample.

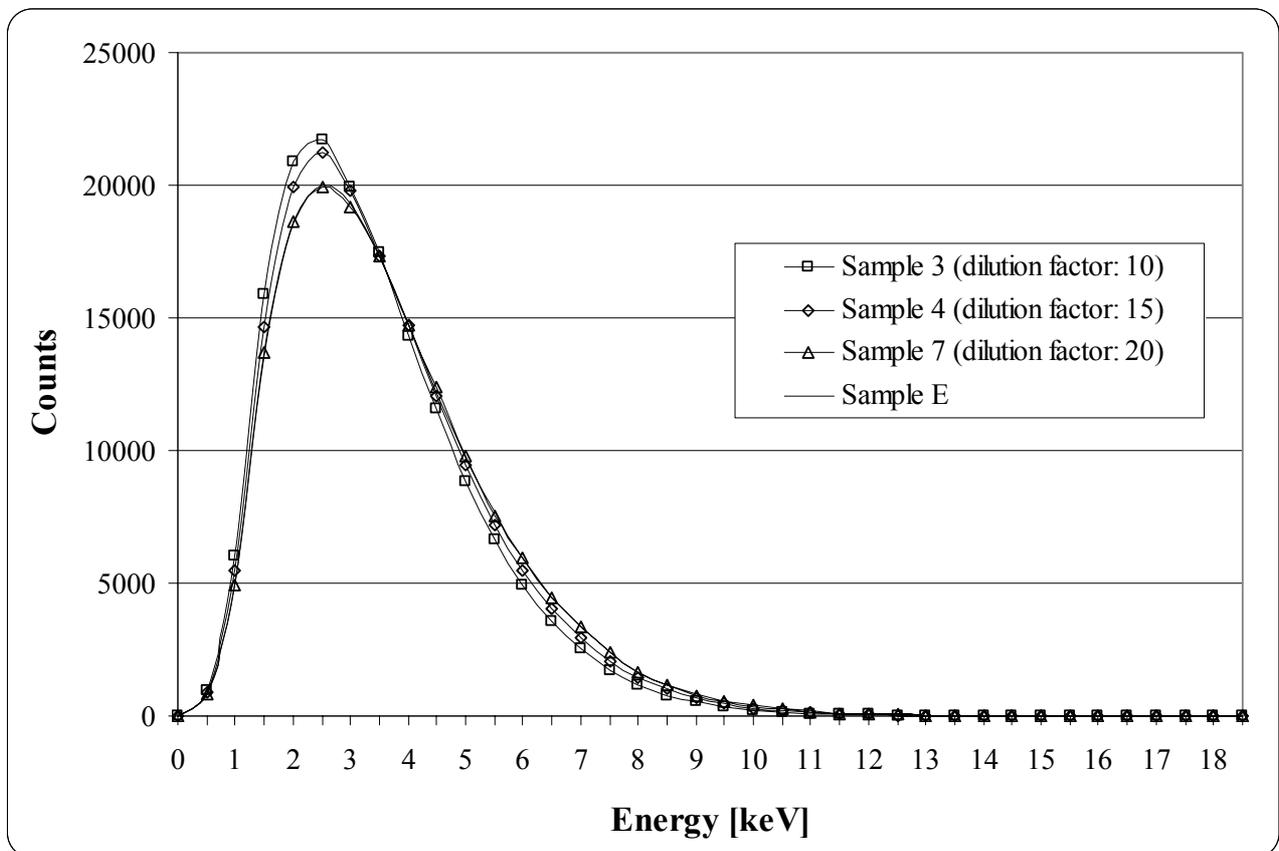


Fig. 3 – Dilution factor effect on beta energy spectra of  $^3\text{H}$  in 3, 4 and 7 samples compared to E sample.

Energy values of  $^3\text{H}$  in the samples 3 (3.44 keV), 4 (3.56 keV) and 7 (3.68 keV) are comparable with 3.70 keV value for  $^3\text{H}$  in standard solution (E sample).

### 1.3. Comparative test for the two types of liquid scintillation cocktails

For sample sets prepared with a dilution factor of 20, sampling a 0.5 mL diluted mineralized solution volume and adding the two liquid scintillation cocktails, it was observed a significant difference between relative deviation values: 9.97 % for Ultima Gold™ AB (sample set no. 6), and 0.15 % for Ultima Gold™ XR (sample set no. 7).

Fig. 4 depicts beta-spectra of  $^3\text{H}$  of the 6, 7 samples compared to the sample E.

The energy spectrum detected from  $^3\text{H}$  in the sample 6 is shifted toward lower values (2.81 keV) compared to 3.68 keV value for the sample 7, and 3.70 keV value for the sample E.

### 1.4. Comparative test for loading factor of the samples

A loading factor comparison test was performed. For no. 4 and no. 5 sample sets prepared with

Ultima Gold™ XR and a dilution factor of 15, the doubling of loading factor (from 0.5 mL to 1 mL) increases both relative deviation and quench grade. Also, for no. 7 and no. 8 sample sets (obtained by applying the same conditions, but with a dilution factor of 20) it is observed the same tendency to increase the relative deviation value, but the counting efficiency is practically equal with E sample set value.

Fig. 5 presents beta-spectra of  $^3\text{H}$  of the 4, 5, 7, 8 samples relative to the sample E.

The results of the experimental tests showed that a dilution factor of 20, followed by sampling of a 0.5 mL diluted mineralization solution volume and homogenization with Ultima Gold™ XR (sample set no. 7) can be used for  $^3\text{H}$  activity evaluation in solubilized graphite samples by microwave acid digestion.

## 2. Assessment of $^3\text{H}$ recovery

The counting efficiencies values of  $^3\text{H}$  in G1 and G2 sample sets were evaluated and are presented in Table 4.

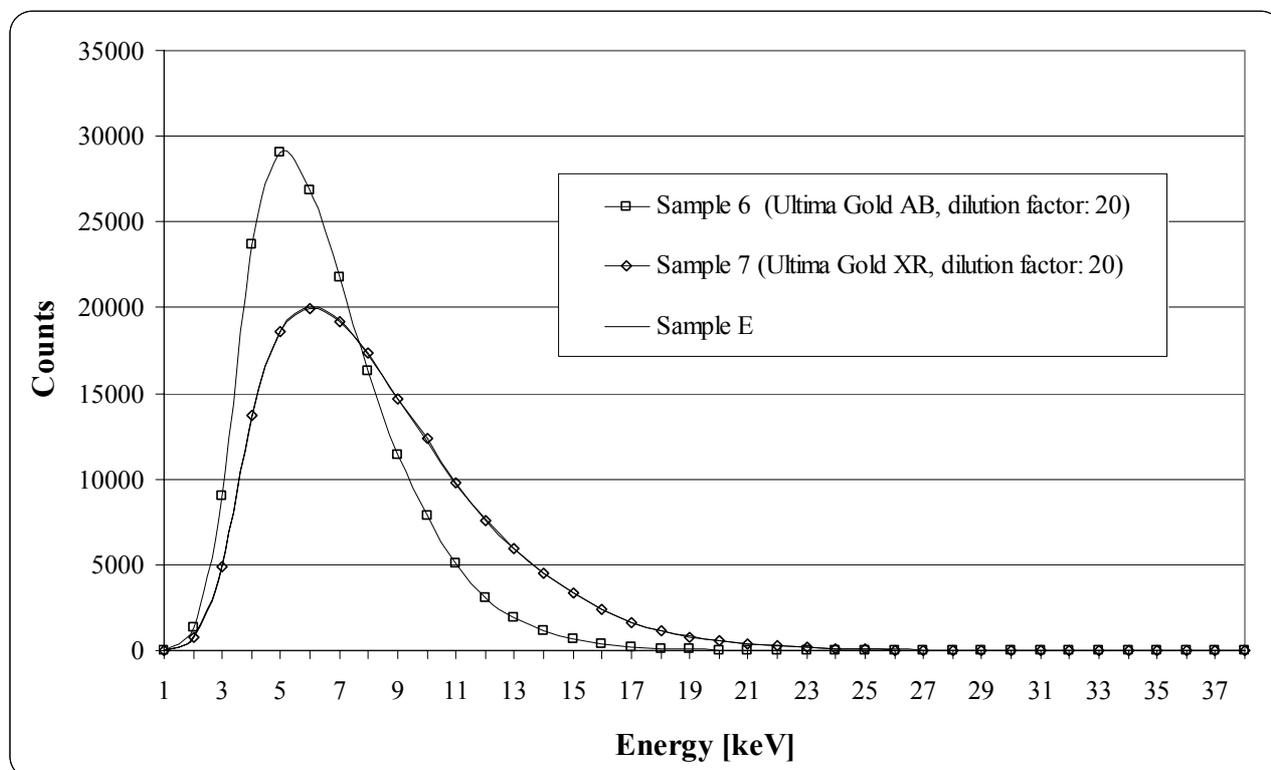


Fig. 4 – Liquid scintillation cocktail type effect on beta energy spectra of  $^3\text{H}$  in 6 and 7 samples compared to E sample.

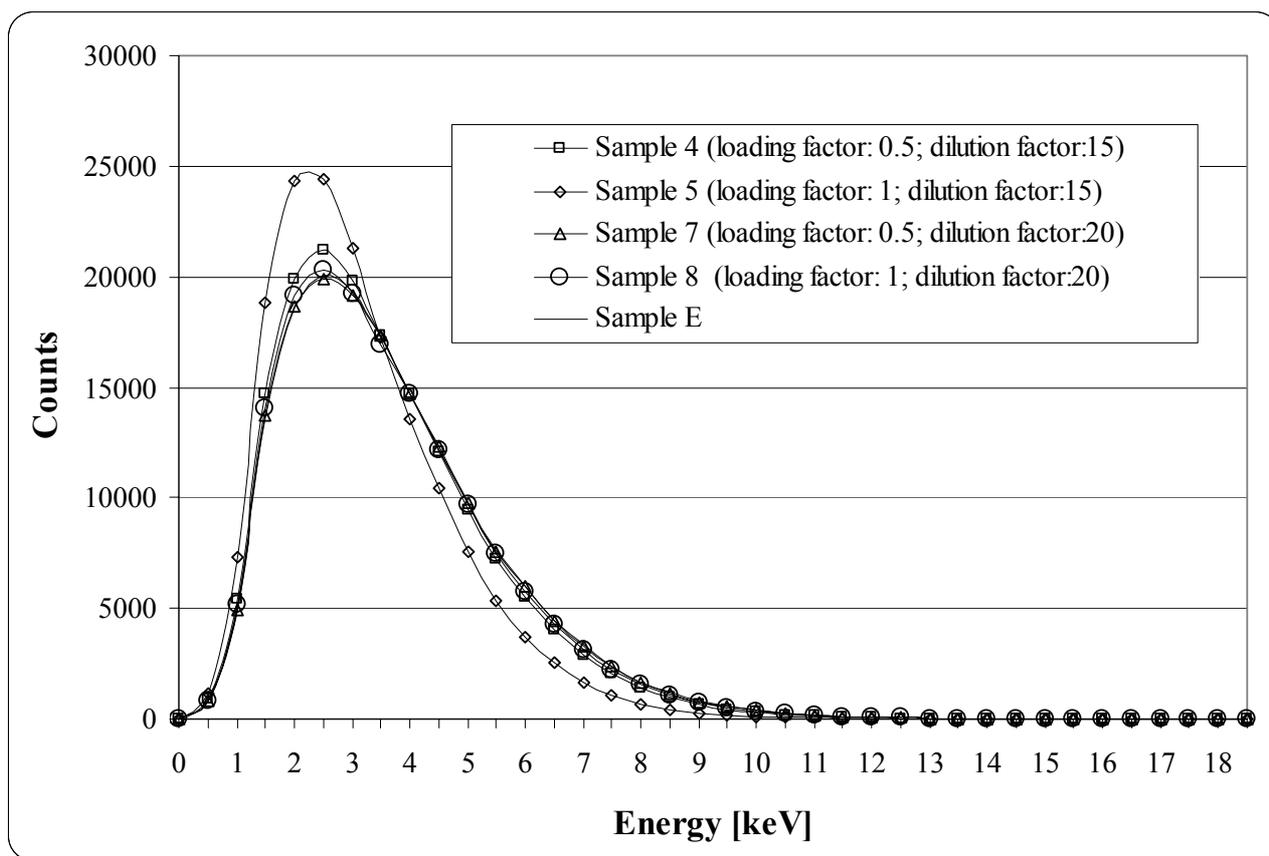


Fig. 5 – Loading factor effect on beta energy spectra of  $^3\text{H}$  in 4, 5, 7 and 8 samples compared to E sample.

Table 4

Counting efficiency values of  $^3\text{H}$  in G1 and G2 samples

Sample code (G1.r), $i=\{1, 2\}$ ; $r=\{1, 2, 3\}$	Counting efficiency [%]
G1.1	$42.91 \pm 0.30$
G1.2	$43.02 \pm 0.30$
G1.3	$42.84 \pm 0.30$
<b>G1 (average of G1.r)</b>	<b><math>42.93 \pm 0.18</math></b>
G2.1	$40.63 \pm 0.29$
G2.2	$41.14 \pm 0.29$
G2.3	$40.58 \pm 0.29$
<b>G2 (average of G2.r)</b>	<b><math>40.78 \pm 0.17</math></b>

The combined uncertainties for the counting efficiency were calculated as in **Evaluation of liquid scintillation cocktail performance** paragraph.

Fig. 6 displays beta-spectra of  $^3\text{H}$  of the G1, G2 samples compared to the sample E.

Energy values of  $^3\text{H}$  in the G1 (3.48 keV) and G2 (3.46 keV) sample sets are comparable with value of 3.70 keV of  $^3\text{H}$  in standard solution (sample set E).

The tritium recovery results from the G1 and G2 analyzed sample sets are presented in Table 5.

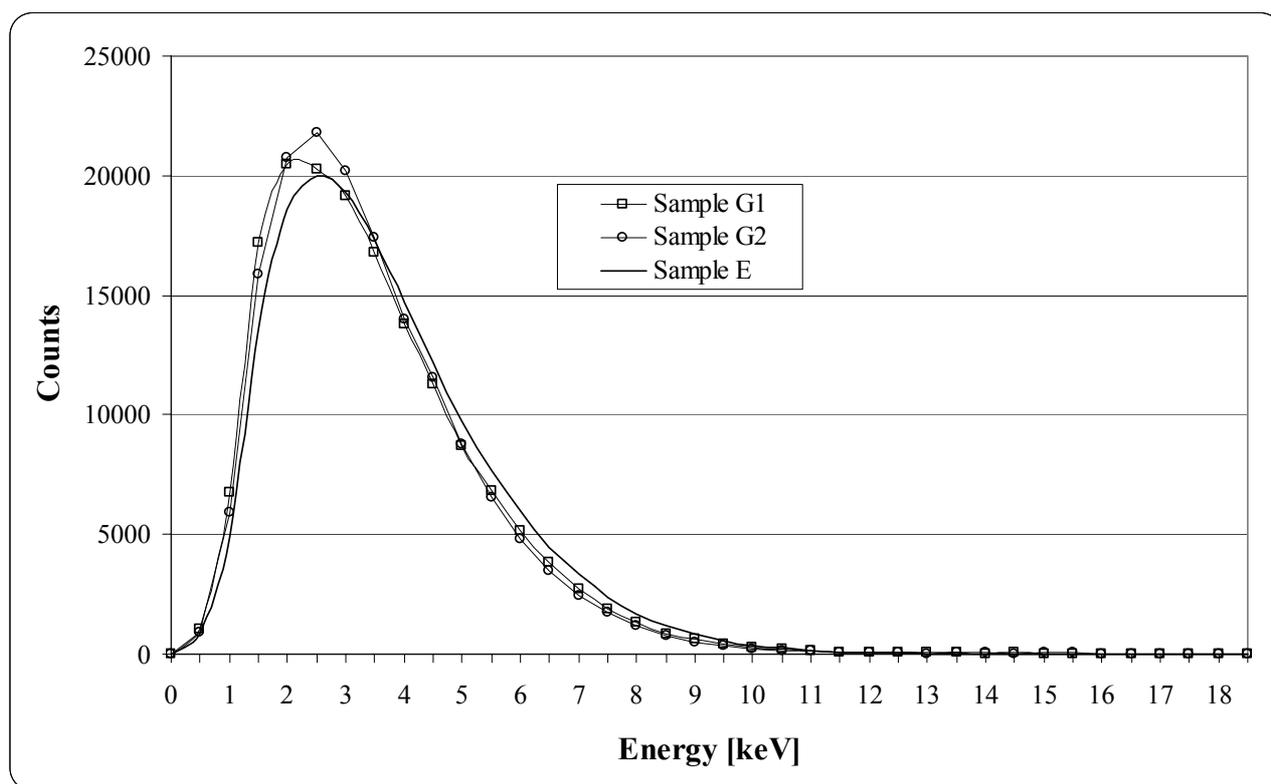


Fig. 6 – Beta energy spectra of  $^3\text{H}$  in G1, G2 samples compared to E sample.

Table 5

Recovery yield of  $^3\text{H}$  after microwave acid digestion

Sample code	Known activity concentration [Bq·L <sup>-1</sup> ]	Measured activity concentration [Bq·L <sup>-1</sup> ]	Recovery [%]
G1	$(8.874 \pm 0.218) \cdot 10^4$	$(8.607 \pm 0.114) \cdot 10^4$	$96.99 \pm 2.71$
G2	$(8.874 \pm 0.218) \cdot 10^4$	$(8.626 \pm 0.114) \cdot 10^4$	$97.20 \pm 2.71$

The known activity concentration values were calculated using the following data: the standard activity value (Table 3), a series of successive dilutions values and a conversion formula from [DPM] to [Bq·L<sup>-1</sup>]. The combined uncertainty of the known activity concentration was accordingly calculated,<sup>22</sup> taking into account the standard uncertainty of the instrumental tools involved in the process (mainly pipettes) as type **B** of uncertainty evaluation (values were not obtained from repeated observations).<sup>21</sup>

The measured activity concentrations were calculated using the converting formula of measured activity value from [DPM] to [Bq·L<sup>-1</sup>]

(Fig. 7). The uncertainties of the measured activity concentrations were combined calculations from type **A** evaluation of measured DPM uncertainties and type **B** evaluation of the uncertainties of volumes transferred by pipette.<sup>22</sup>

Fig. 7 presents the Ishikawa (or fishbone) cause and effect diagram<sup>23</sup> for the analysis of uncertainty sources contributions on measured activity concentration.

The combining rule of standard uncertainty of a quotient expression was used to obtain the recovery process combined uncertainty.<sup>22</sup>

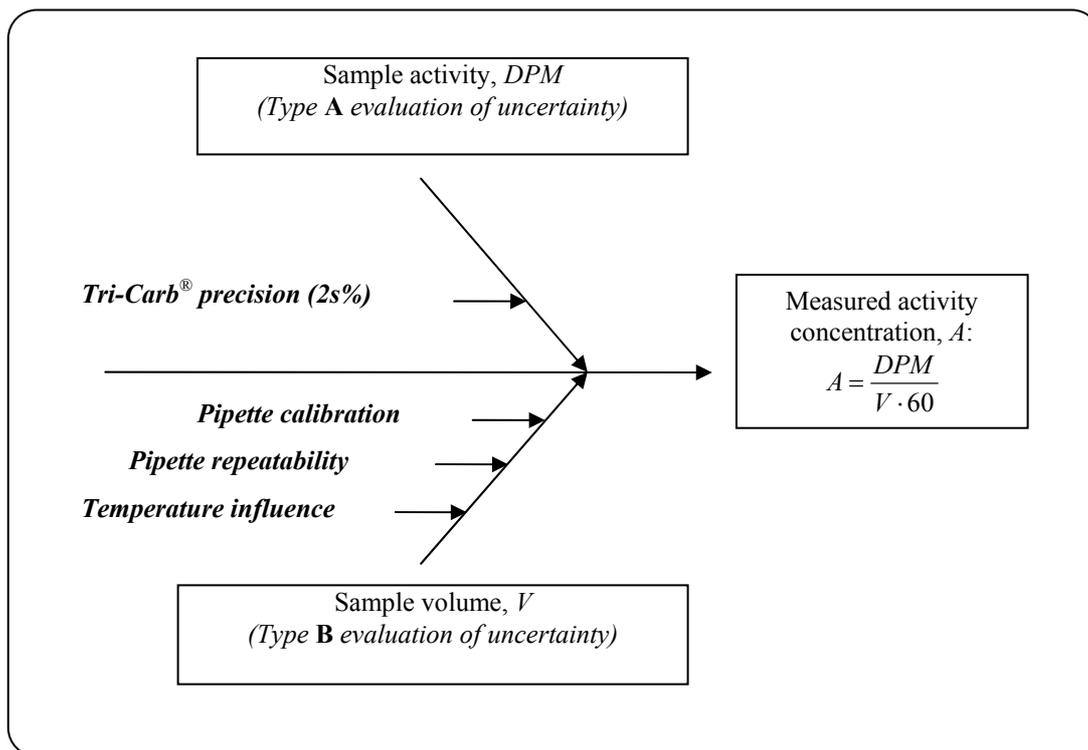


Fig. 7 – Cause and effect diagram for measured activity concentration.

### 3. Detection limit of the method for tritium

The method of Currie<sup>24</sup> defines the lower limit of detection as Eq. (1).

$$L_D = 1.645^2 + 2 \cdot 1.645 \sqrt{R_B \cdot T_S \cdot \frac{T_B + T_S}{T_B}} \quad (1)$$

where  $L_D$  is lower limit of detection (counts);  $R_B$  is background count rate (cpm);  $T_B$  is background count time (minutes) and  $T_S$  is sample count time (minutes).

The measuring time for the blank sample was 90 minutes. This time was chosen as the count termination parameter for the blank sample, and the count rate obtained was  $5.00 \pm 0.88$  cpm with a 2s% reported statistical precision of 17.51 %. The G1.2 sample count time was of 136.52 minutes, where a 2s% statistical precision of 0.5 % was used as a count termination parameter for the sample counting. The G1.2 sample count time was chosen as the minimum count time from all the 6 counting times of the samples with G1.r codes (the detrimental case for lower limit of detection calculation). Using these values,  $L_D$  is calculated to be  $139.07 \pm 11.94$  counts.

For a 0.5 mL sample volume and a maximum counting efficiency of  $43.02 \pm 0.30$  % (i.e. G1.2 sample, the detrimental case for the estimation),

the minimum detectable activity calculated as in Eq. (2) is  $78.93 \pm 6.87$  Bq·L<sup>-1</sup>.

$$MDA = \frac{L_D}{Ef \cdot V \cdot 60 \cdot T} \quad (2)$$

where  $MDA$  is minimum detectable activity (Bq·L<sup>-1</sup>);  $L_D$  is lower limit of detection (counts);  $Ef$  is counting efficiency;  $V$  is sample volume (L); 60 is time conversion factor from minutes to seconds and  $T$  is measuring time (minutes).

If the measuring time is much smaller than the half-life of the measured nuclide, it may be approximated with the sample count time,<sup>24</sup> so for the calculated value of  $MDA$ , the G1.2 sample count time of 136.52 minutes was used for the  $T$  value in Eq. (2).

As activity concentration of <sup>3</sup>H in irradiated graphite samples from thermal column of TRIGA reactor is spanning ( $10^3 \dots 10^4$ ) Bq·g<sup>-1</sup> values (estimated on the base of the combustion technique using a sample oxidizer),<sup>10</sup> for a 0.05 g amount of graphite per digestion vessel the equivalent of (50...500) Bq radioactivity is obtained, or equivalent of  $(2 \dots 20) \cdot 10^3$  Bq·L<sup>-1</sup> for a solution volume of 25 mL per digestion vessel.

Therefore, the developed method presents a high level of sensitivity for tritium radioactivity determination in graphite waste mineralized by microwave acid digestion.

## CONCLUSIONS

The main purpose of this paper was to develop both a procedure for mineralization of graphite samples and a preparation method of solubilized graphite samples for tritium measurement.

The mineralization procedure of the graphite samples was efficiently achieved by using a closed system formed by a microwave vessel system and a mixture of concentrated acids.

The developed methodology for solubilized samples preparation allowed: good counting efficiency for tritium evaluation (over 40 %); high level of tritium recovery (around 97 %); and assessment of the minimum detectable activity for investigated radionuclide (just about 80 Bq·L<sup>-1</sup>).

The proposed conditions both for graphite chemical dissolution (using microwave technology) and for preparation of solubilized graphite samples for LSC can be applied to the irradiated graphite samples collected from the thermal column of TRIGA reactor.

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