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**Influence of acid leaching to  
determine natural radionuclides  
in sand samples by alpha  
spectrometry**

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## **ABSTRACT**

Dissolution of sand samples in order to determine natural radionuclides as Uranium and Polonium is an essential step in alpha spectrometry analyses. The aim of this work has been to analyse which digestion method for alpha spectrometry measurement of uranium and polonium radioisotopes in sand samples from Las Canteras beach (Gran Canaria) is the most appropriate. This study was carried out by varying the digestion time of sand and reference samples in the oven and also by performing a microwave digestion. A change in the triplet of acids used on the microwave digestion was also evaluated. The polonium and uranium radioisotopes were extracted by a radiochemical separation method and measured by alpha-spectrometry. To provide an optimal evaluation of the most efficiency digestion method applied to the reference and the sand samples, the Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) proficiency test was used.

**Keywords:** radionuclides, digestion, proficiency test, alpha spectrometry.

## 1. INTRODUCTION

Alpha spectrometry is the most widely used technique to obtain the contents of uranium and polonium radionuclides concentrations in a variety of samples (soil, water, plants) both environmental and industrial samples (Lozano et al., 2010). Its use, however, requires specific conditions such as the elimination of other interfering alpha emitters. This means that in most cases the samples must be processed physically or chemically before the spectrometric measurements. (Tomé et al., 2002).

Usually, the alpha spectrometry technique for solid samples (biota, soil, sediments, sands, et) means different stages: 1) sample pre-treatment, mainly making a digestion of the sample with strong acids that break the mineral structure of the solids; 2) isolation by phases of polonium and uranium (radiochemical analysis); and 3) measurement of alpha emitters with a spectrometer (Lozano et al., 2010).

According to Marlap, (2004) the most important step in the determination of radionuclides in solid samples is the dissolution of the samples. It is necessary to dissolve it completely in order to avoid losing activity due to these isotopes becoming trapped in the insoluble residue. The most common method to dissolve solid samples to extract analytes is acid digestion. The traditional acid digestion is performed in teflon capsules, with acid mixtures and high temperature in an oven. This method has a series of advantages and disadvantages. With regard to the advantages, these containers are coated with Teflon which can contain strong acids at very high temperatures, such as HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub> or aqua regia (HNO<sub>3</sub> and HCl), and it is also possible to use HF. In addition, this method, being carried out in closed Teflon vessels, favors resistance to cross contamination between samples. Also, the dissolution of the sample is obtained without losing volatile elements. However, there are certain drawbacks. This type of digestion is slow, important volumes of acids are consumed and generally does not give good results if the sample contains a highly insoluble resistance.

On the other hand, microwave digestion has practically replaced previous conventional methods and is now used for the preparation of many types of sample matrices. The advantages of this technique for elementary analysis have been studied extensively (Chevallier et al., 2015; Kingston et al., 2017; Ozden et al., 2017). The acid attack in microwave pressure reactors is much faster and more effective and in a few minutes the result is available, being possible to make several digestions in one day varying the acid mixtures used, the temperature and the digestion times, unlike in traditional digestion, which takes hours or even days, and can only be repeated until the samples are completely dissolved. Therefore, it could be seen as a more effective method for the preparation of solid samples (Abu-Samra et al., 1975; ASTM D7876-13, 2018; Chakraborty, 1996; de la Torre Pérez et al., 2013; Guirguis et al., 2015; Kingston and Jassie, 1988; Smith and Arsenault, 1996)

With regard to radiochemical analysis and based on Crespo (1996), it is necessary to put through the marine environmental samples to several radiochemical treatments to isolate the radionuclides of interest ( $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$ ) that can involve losses of the sample. For this reason, before beginning any process in which these losses may occur, a known quantity of a certain radionuclide that is not present in the sample and has the same physicochemical properties as the radionuclides that are determined should be added to the original sample. This radionuclide that is added is known as a tracer. From the relationship between the amount of tracer detected in the measurement and the quantity initially added, the overall performance of all the manipulations to which the sample has been subjected is determined.

There are several radiochemical methods for extracting the radionuclides in soil samples for measurement by alpha spectrometry like solvent extraction, selective precipitation or ion exchange resins. Moreover, in recent years new extractants have been developed for the chromatography of partitions technique, specifically for the determination of actinides (Marabini and Serdeiro, 2003). One of the most used methods for the extraction of radionuclides in environmental samples is the liquid-liquid extraction (LLE). This technique is based on the chemical affinity of an analyte for one of two essentially immiscible solvents, one aqueous and one organic. The analyte is positioned in one of the two phases and can be extracted from its matrix. This mixture must be produced by agitation. This method is selective for radioisotopes such as uranium and polonium. This extraction with organic solvent for the selective and sequential isolation of the radioisotopes of polonium and uranium, is based on the method initially developed by Holm and Fukai (1977), in which the organic phase is formed by tributyl-n-phosphate (TBP) and xylene. It should be added that one of the critical stages for uranium and polonium determination is the chemical separation of the radionuclides from the matrix through the LLE (Díaz Francés, 2014; Maese, 2016; Prasada et al., 2006).

According to the work of Hallstadius (1984) and Garcia Tenorio et al., (1986), electrodeposition, the final phase of the radiochemical analysis, is a type of preparation of sources of measurement by alpha spectrometry. It is based on the forced deposition by electrolysis of the radionuclides of interest on a surface, which can be a plate of stainless steel or silver. The radionuclides are in a solution that must be previously conditioned. The induction of current by the solution is produced creating a potential difference between the metal planchette (cathode) and a platinum wire (anode). The current causes the deposition of the radioisotopes of interest on the surface of the planchette that is exposed to the solution (electrolyte) in the electrodeposition cell. This critical stage of deposition of uranium should provide a very thin deposit for measurement. The thickness of this deposit is essential for a high resolution of the subsequent measurement peaks in the alpha spectrometer. Apart from electrodeposition,

there is another type of preparation of sources for measurement by alpha spectrometry. This is mechanical self-deposition, which is used for  $^{210}\text{Po}$  determination. The self-deposition is used on a material that has a lower redox potential, such as Ag, Cu or Ni. The most used method was developed by Flynn, (1968) and is based on the spontaneous deposition of  $^{210}\text{Po}$  on silver planchets in different conditions with high yields, although there are also methods for the preparation of measurement sources in copper planchets (Aslani et al., 2005). However, according to Díaz Francés, (2014) the advantage of using silver planchets relies on the fact that the deposition is selective for this element, not producing the deposition of other alpha emitters that could interfere and that were present in the carrier solution, of polonium (as for example the Ra).

When working with environmental samples, it is impossible to know exactly at which level the radionuclides are integrated in the sample, so it is hard to know which type of digestion is more appropriated for treating each sample in order to determine  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$  by alpha spectrometry. In this framework the main objective of this work is to study which digestion method for alpha spectrometry measurement of uranium and polonium radioisotopes in sand samples from Las Canteras beach (Gran Canaria) is the most suitable. This evaluation will be carried out by varying the hours of digestion samples. Microwave digestions will also be carried out to compare the results obtained in both methods. After the digestion, a radiochemical separation method of actinides will be performed to isolate polonium and uranium to finally measure the samples in alpha spectrometry. To provide an optimal evaluation the Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) proficiency test will be used. These results are presented in part 3 and the conclusions are in section 4.

## **2. EXPERIMENTAL**

### *2.1.- Sampling*

Forty-eight samples were prepared for this study, nineteen were reference material prepared by reference laboratories and with activity concentrations of different radionuclides well-known and twenty-nine were sand samples from Las Canteras beach.

The IAEA-RGU-1 reference material is a dilution of a uranium ore, BL-5 (7.09% U), and a thorium ore, OKA-2 (2.89% Th, 219  $\mu\text{g}$  U/g), with floated silica powder of similar grain size distribution and prepared on behalf of the International Atomic Energy Agency (IAEA) by the Canada Centre for Mineral and Energy Technology. BL-5 has been certified for uranium,  $^{226}\text{Ra}$  and  $^{210}\text{Pb}$  confirming that it is in radioactive equilibrium. The activity concentrations values of the  $^{238}\text{U}$  and  $^{235}\text{U}$  are listed in Table 1.

| <i>Radionuclides</i> | <i>Value</i> | <i>95% C. I.</i> |
|----------------------|--------------|------------------|
| $^{238}\text{U}$     | 4940         | 4910 - 4970      |
| $^{235}\text{U}$     | 228          | 226 - 230        |

Table 1.- Activity concentrations (Bq/kg) of uranium radioisotopes in RGU-1 reference sample. C.I: Confidence Interval.

The IAEA-447 is a sample of the moss-soil, also Certified Reference Material, that is characterized on the basis of results reported by the IAEA Terrestrial Environment Laboratory in Seibersdorf, Austria, (Shakhashiro et al., 2012). The material was analysed by two expert laboratories; Radioanalytical Laboratory, Food and Feed Safety Division in Budapest, Hungary, and Laboratory of Radiological Measurements, Jozef Stefan Institute in Ljubljana, Slovenia. The activity concentrations of radionuclides are shown in Table 2.

| <i>Radionuclides</i> | <i>Value</i> | <i>Uncertainty</i> |
|----------------------|--------------|--------------------|
| $^{238}\text{U}$     | 22.2         | 0.8                |
| $^{235}\text{U}^*$   | 1.02         | 0.04               |
| $^{234}\text{U}$     | 21.8         | 0.8                |
| $^{210}\text{Po}$    | 423          | 10                 |

Table 2.- Activity concentrations (Bq/kg) of radionuclides in IAEA-447 reference sample. \* was calculated from the certified values and the natural abundance.

The IAEA-448, a sample of contaminated bulk soil, was collected in September 2007 from Syrian oil field by staff of the Atomic Energy Commission of Syria, an IAEA collaborating centre. The activities concentrations of radionuclides are shown in Table 3.

| <i>Radionuclides</i> | <i>Value</i> | <i>Uncertainty</i> |
|----------------------|--------------|--------------------|
| $^{238}\text{U}$     | 49.2         | 0.9                |
| $^{235}\text{U}$     | 2.3          | 0.1                |

Table 3.- Activity concentrations (Bq/kg) of radionuclides in IAEA-448 reference sample.

The remaining samples were environmental samples, sands, collected in different parts of Las Canteras beach in October 2016 and March 2018. The sands of the samples used in this work correspond to three zones of the beach with different composition. The regions are showed in Figure 1 and the samples are listed in Table 4.





Figure 1.- Division of Las Canteras beach and location of the sand samples used in this work.

| <i>Sample</i> | <i>Type of sample</i> | <i>Zone of the Las Canteras beach</i> |
|---------------|-----------------------|---------------------------------------|
| PLC16_5.2     |                       | Southern arch                         |
| PLC16_5.3     |                       | Southern arch                         |
| PLC18_3.2     | Sand                  | Southern arch                         |
| PLC18_3.5     |                       | Central arch                          |
| PLC18_3.10    |                       | Northern arch                         |
| IAEA-447      | Reference materials   | -                                     |
| IAEA-448      |                       | -                                     |
| IAEA-RGU-1    |                       | -                                     |

Table 4.- Reference and sand samples of the Las Canteras beach of 2016 and 2018 campaigns.

## 2.2.- Apparatus

The detection system used for the measurements of the analyzed alpha samples in this work is a spectrometry system, Alpha Analyst model, manufactured by Canberra. This system consists of 12 independent spectrometric units (Figure 2a), each with an ion-implanted silicon detector (PIP) (Figure 2b), Model PD-450.18 AM with 450 mm<sup>2</sup> active area. The spectrometer is connected to a vacuum pump to achieve the vacuum conditions necessary for alpha particles to reach the detector without losing energy and optimal measurement is made in the 12 chambers. The range of admitted values for the vacuum pressure is between 0.05 - 0.5 Torr (Garcia Tenorio et al., 1986).

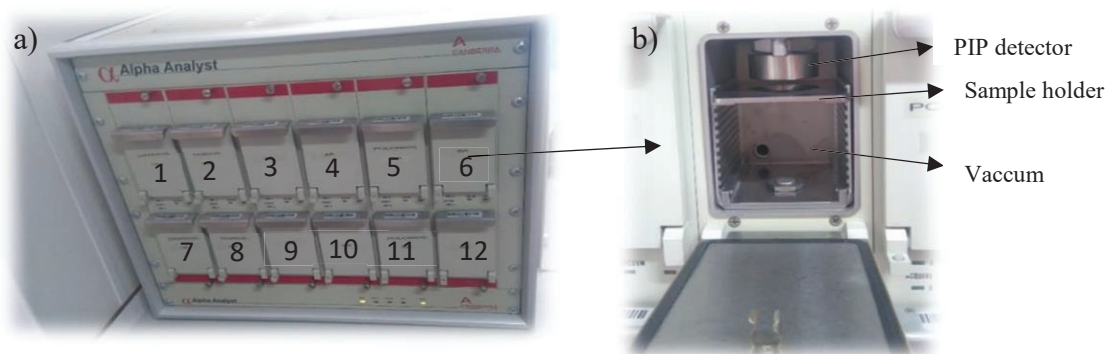


Figure 2.-a) Front of the spectrometer with 12 independent spectrometric units and b) Open chamber where the detector can be seen.

In this work, the detectors were energy and efficiency calibrated. First, in the energy-calibration using traceable mixed standard alpha source containing  $^{233}\text{U} + ^{239}\text{Pu} + ^{241}\text{Am}$  (FRC2013-317), provided by CIEMAT (Centro de Investigaciones Medioambientales y Tecnológicas, Madrid, Spain) (Table 5). Secondly, the efficiency-calibration of the detector, which is the ratio between the number of particles detected and the number of particles emitted, for each detector used in this work, is shown in Table 6.

| <i>Radionuclide</i> | <i>Energy (keV)</i> | <i>Intensity (%)</i> |
|---------------------|---------------------|----------------------|
| $^{233}\text{U}$    | 4905.5              | 100                  |
| $^{239}\text{Pu}$   | 5244.5              | 100                  |
| $^{241}\text{Am}$   | 5637.8              | 100                  |

Table 5.- Characteristics of the source of alpha emitters used.

The samples were generally measured at about 5 mm from the detector and during 345,600 s to get appropriate minimum detectable activities (MDA) of 0.1 mBq/L (U) and 0.4 mBq/L (Po) (Tejera et al., 2018).

| <i>Detector</i> | <i>Efficiency (%)</i> | <i>Detector</i> | <i>Efficiency (%)</i> |
|-----------------|-----------------------|-----------------|-----------------------|
| 1A              | 25.54                 | 5A              | 21.32                 |
| 1B              | 22.93                 | 5B              | 24.82                 |
| 3A              | 23.11                 | 6A              | 20.74                 |
| 3B              | 25.21                 | 6B              | 22.82                 |

Table 6. Counting efficiency in the eight chambers in the alpha spectrometry system.

### 2.3.- Analytical technique

The analysis technique developed in this work is described next. This basically includes the preparation of the sample (digestion), radiochemical separation of radionuclides and the counting procedure. Figure 3 shows a diagram where all the steps of the analytical procedure are summarized.

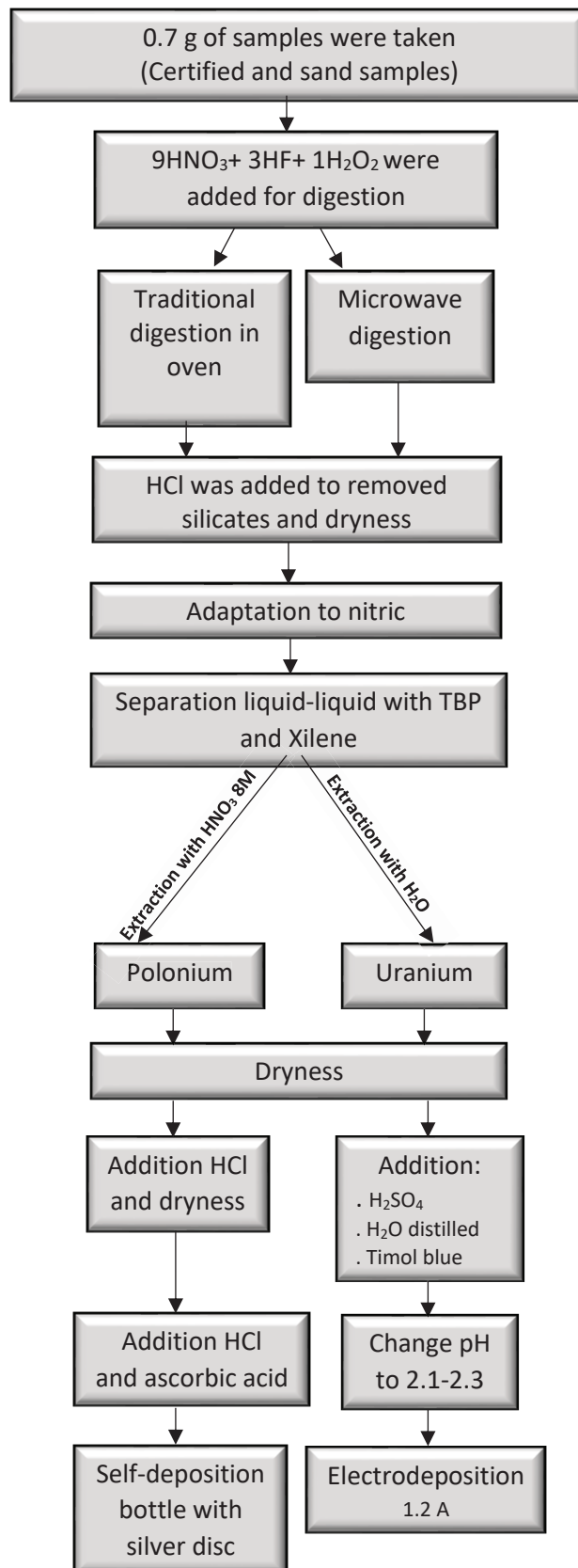


Figure.3.- Flow chart of the experimental procedure used in this work for preparation of uranium and polonium sources from solid samples.

### 2.3.1.- Preparation of the sample

First, sand samples (Figure 4) were taken to the laboratory and were oven dried at 90°C for 24 hours. After they were screened through a 1 mm mesh size sieve and milled in Agatha vessel in order to reduce the sand to fine powder. Considering that the certified reference materials were as dashes they did not have to be milled.

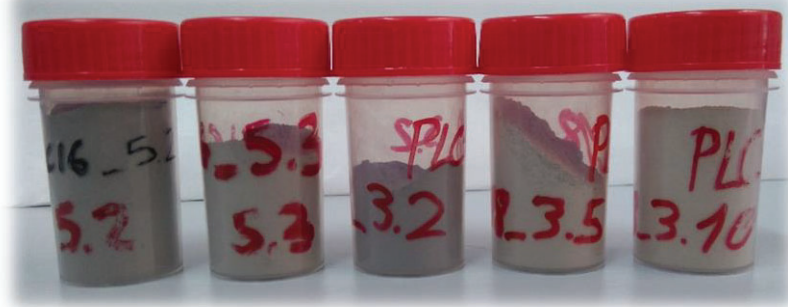


Figure 4.- Sand samples of the Las Canteras beach

The second step is the complete dissolution of the sample. Two processes of digestion have been developed in this work: a classic digestion in Teflon container and a microwave digestion.



Figure 5.- a) Three aliquots in Savillex vessel (24, 48, and 72 hours at 90 °C and b) sample in microwave vessels.

#### a) Acid leaching method

For all the prepared samples, the same amount as well as the same mixture of acids of sample was used. Three aliquots of about 0.7 g of weight were taken and put on a Savillex glass with concentrated nitric (69%) and hydrofluoric (48%) acids and hydrogen peroxide (33%) in proportion 9:3:1. To test the efficiency of digestion time in determining alphas emitters on sand samples, each aliquot was subjected to digestion for 24 (aliquot A), 48 (aliquot B) and 72 (aliquot C) hours (Figure 5a). The radiochemical yields for the uranium and polonium analyses by alpha spectrometry in these samples were determined by using  $^{232}\text{U}$  and  $^{209}\text{Po}$  tracers. After the digestion time,

each sample is transferred to a Teflon glass, HCl (37%) is added and the sample is brought to dryness at a temperature below 90°C to avoid polonium volatilization.

*b) Microwave digestion*

The second procedure for the digestion was based on the use of Titan MPS microwave sample preparation system. In this case, the environmental digestion method for soil described in (EPA, 2007; Mola et al., 2013) was selected. As we discussed earlier, the same amount of sample and tracers as in the previous case was also used in this procedure. The digestion conditions are shown in Table 7.

| <i>Steps</i> | <i>Temperature<br/>°C</i> | <i>Pressure<br/>limit (bar)</i> | <i>Ramp time<br/>(min)</i> | <i>Hold time<br/>(min)</i> | <i>Power<br/>(%)</i> |
|--------------|---------------------------|---------------------------------|----------------------------|----------------------------|----------------------|
| 1            | 175                       | 60                              | 5                          | 5                          | 90                   |
| 2            | 50                        | 30                              | 1                          | 10                         | 0                    |

Table 7.- Conditions of microwave digestion of solids samples modified from Perkin, 2016.

Once finished, samples (figure 5b) were transferred to Teflon glass and HCl (37%) was added and the sample as aforementioned. In order to verify which is the most effective for the sand samples different methods and digestion times were tested. A new triplet of acids was used too as HF:HCl:HNO<sub>3</sub> (10:5:2.5), and several samples, sand and certified, were digested with these new acids.

*2.3.2.- Radiochemical separation of radionuclides*

After digestion in oven or microwave and HCl process had done, 10 ml HNO<sub>3</sub> (69%) were added to the sample and it was transferred to a beaker, where it was evaporated to dryness, at 80°C maximum. Finally, the residue was dissolved with 10 ml of HNO<sub>3</sub> (8M) and transferred to a clean funnel for extraction. Then, for extracting U, Th and Po, tri-n-butyl phosphate (TBP) and xylene were added (Mola et al., 2013)

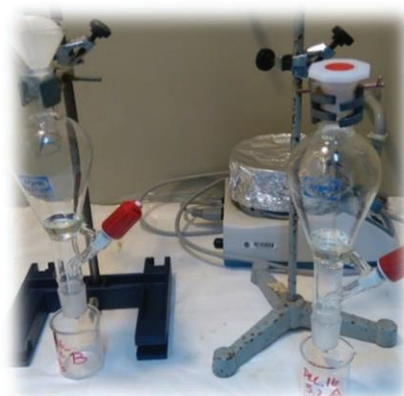


Figure 6.- Samples in funnels were settled

*a) Polonium*

After the sample was transferred to a funnel, the beaker was washed with 5 ml of HNO<sub>3</sub> (8M) and the solution was added to the funnel too. Then, 5ml to TBP tributyl phosphate, an organic dissolvent, was added (Figure 6). The funnel was agitated for 10 minutes and then settled for another 10 minutes without cover. When the different phases could be seen easily they were separated. Then 10 ml of HNO<sub>3</sub> 8M were added



to the funnels and previous two steps were repeated. After two extractions the polonium of each samples were taken to self-deposition phase.

#### *a.1) Self-deposition Polonium*

$^{210}\text{Po}$  solution was evaporated to dryness. Then, the residue was dissolved with HCl, and 50 mg of ascorbic acid was added in order to reduce the  $\text{Fe}^{3+}$  into  $\text{Fe}^{2+}$ . If some particles were observed the solution was filtered and transferred to a self-deposition bottle. Finally, polonium was spontaneously deposited overnight onto a silver disc with mechanic stirring at room temperature.

#### *b) Uranium*

For Uranium radionuclide extraction, 15 ml of distilled water were added to the organic phase where the uranium was retained after the previous extractions. The uranium was back-extracted with water, the funnel was agitated and settled in the same way as the polonium and the procedure was repeated two times as well. Finally, the uranium was extracted and taken to electrodeposition phase.

#### *b.1 Electrodeposition Uranium*

After, the sample was evaporated until 1 or 2 ml, 1ml of  $\text{Na}_2\text{SO}_4$  was added to fix the uranium radionuclides and was evaporated to dryness. The residue was dissolved with 0.3 ml of  $\text{H}_2\text{SO}_4$  (98%) and 4 ml of distilled water. Finally, 4 drops of Thymol blue indicator were added and pH was adjusted to 2.1-2.3 with  $\text{NH}_3$  (5M). The electrodeposition was performed in an electrodeposition cell (Figure 7) during 1 hour at 1.2 A of electric current. The distance between cathode and anode was 4-5 mm.

At this point, it is also important to note that the electrodeposition is a critical stage. It is essential for a high resolution of the spectrum peaks that a very thin deposit is obtained since if the deposit is too thick the alpha particles cannot escape and do not reach the detector (Hallstadius, 1984). In Figure 8 two planchets, one with good deposition (a) and another with bad deposition (b) can be seen.

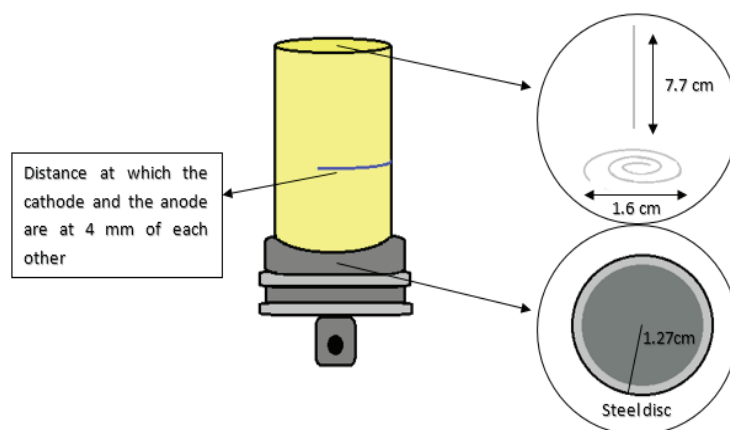


Figure 7.- Sketch of electrodeposition cell with steel disc and platinum wire

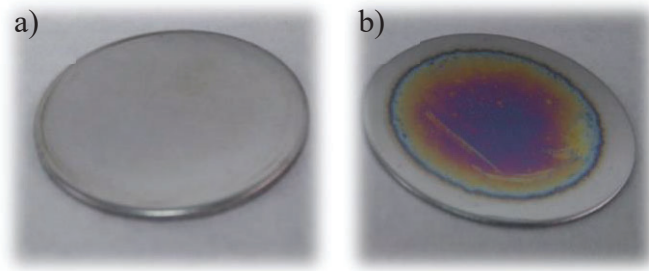


Figure 8.- a) Clean steel disc and b) steel disc with uranium deposition of IAEA-RGU-1.

#### 2.4. Activity calculation

The specific activity of the isotope is calculated by following the next expression:

$$A_{el} = \frac{N_I}{\varepsilon t I r m} = \frac{N_I}{N_T} \frac{I_T}{I_I} \frac{1}{m} \quad (1)$$

where  $N$  is number of net counts of the radioisotope; (number of net counts less background counts of the radioisotope);  $\varepsilon$  is the detector efficiency;  $t$  is the counting time;  $I$  is the intensity of the detected radioactive emission;  $r$  is the measurement yield and  $m$  is the sample mass. The subscripts  $I$  and  $T$  indicate sample and tracer respectively.

The uncertain associated to this specific activity is:

$$u(A) = A_{el} \sqrt{\frac{1}{N_I} + \frac{1}{N_T}} \quad (2)$$

where  $A_{el}$  is the activity calculated,  $N_I$  is the net counts of the sample and  $N_T$  represents the net counts of the tracer.

#### 2.5.- Performance criteria

In order to provide an optimal evaluation of the analytical performance of the digestion times applied to both the reference and the sand samples, the Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) proficiency test was used according to IAEA (2011) and modified by Osvath et al., (2016) and Shakhashiro et al., (2012). The ALMERA network consists of 166 laboratories representing 87 countries. One of the goals of this proficiency test is to provide accurate radionuclide analysis in environmental samples, to monitor and demonstrate improvements in the accuracy (trueness and precision) of the method and the international comparability of measurements of radionuclides in the environmental

samples. (IAEA / AL / 164, 2006). To do so, the network laboratories use sets of different samples (typically encountered in environmental and food monitoring laboratories) and after the radiological analysis, their results are discussed in the intercomparing meetings. Based on this, different statistical parameters were calculated (IAEA 2011) to select the best digestion method for our sand samples. These parameters are: Relative Bias (RB), the  $U_{test}$  and the Precision Score ( $P_{Score}$ ).

The RB between radionuclide and reference value is calculated and expressed as a percentage following the next equation:

$$RB(\%) = \frac{value_{Radionuclide} - value_{reference}}{value_{reference}} \times 100 \quad (3)$$

The value of the  $U_{test}$  is calculated according to next expression:

$$U_{test} = \frac{|value_{Reference} - value_{Radionuclide}|}{\sqrt{u_{Reference}^2 + u_{Radionuclide}^2}} \quad (4)$$

For this proficiency test the limiting value for the  $U_{test}$  parameter to determine if a result passes the test is 2.58 for a level of probability at 99% ( $U_{test} < 2.58$ ).

The  $P_{score}$  is calculated as:

$$P_{Score} = \sqrt{\left(\frac{u_{Reference}}{value_{Reference}}\right)^2 + \left(\frac{u_{Radionuclide}}{value_{Radionuclide}}\right)^2} \quad (5)$$

The results were associated as 'Accepted' when  $P_{Score} \leq$  Limit of Acceptable Precision (LAP). Table 8 summarizes the criteria for each parameter and for each radionuclide used in this work.

| Radionuclide      | Relative Bias (RB) | U-test | Precision Score ( $P_{Score}$ ) |       |
|-------------------|--------------------|--------|---------------------------------|-------|
|                   |                    |        | LAP                             | MAP   |
| $^{238}\text{U}$  | RB $\leq$ MAB      | < 2,58 | 20-35                           | 20-35 |
| $^{235}\text{U}$  |                    |        | 20-35                           | 20-35 |
| $^{234}\text{U}$  |                    |        | 20-35                           | 20-35 |
| $^{210}\text{Po}$ |                    |        | 20-35                           | 20-35 |

Table 8.- Table of critical values for the radionuclides according to IAEA (2011) and Osvath et al., (2016)

In the final evaluation, both accuracy and precision scores are combined. For effective results, the "accepted" score must be obtaining in both criteria. Obviously, if the "Not Accepted" score is obtained for both accuracy and precision, the final score will also be "Not Accepted". In cases where either precision or accuracy is "Not



Accepted", the reported RB is compared to the Maximum Acceptable Bias (MAB) (IAEA 2011) and two possibilities can be presented:

- a) If  $RB \leq MAB$ , the final score will be 'Accepted with warning'. The 'Warning' will reflect two situations. The first will be a result with a small uncertainty; however, its bias is still within MAB. The second one appears when a result is reported close to the value of the assigned property, but the associated uncertainty is large.
- b) If  $RB > MAB$ , the result will be 'Not accepted'.

The MAB values used in the evaluation of all radionuclides are listed in Table 8 (IAEA 2011).

This method has been modified by Shkhashiro et al., (2012) and Osvath et al., (2016). Figure 9 represents the flow diagrams of the evaluations of proficiency test results proposed by those authors. These two modified methods are those that we are going to use for the validation of the most efficient digestion method in chapter 4.

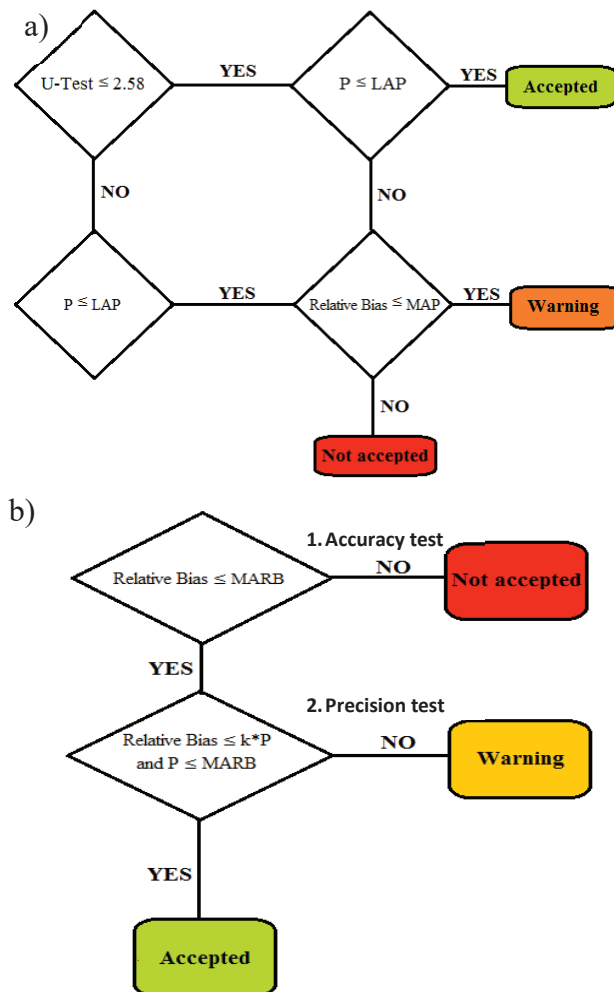


Figure 9.- Proficiency tests according to a) Shkhashiro et al., (2012) and b) Osvath et al., (2016).

### 3. RESULTS

#### 3.1 Activity concentrations of $^{238}\text{U}$ , $^{235}\text{U}$ , $^{234}\text{U}$ and $^{210}\text{Po}$ in sand samples

The activity concentrations of  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$  obtained for each digestion method in all sand samples are shown in Figure 10. These data were obtained by alpha spectrometry and correspond to the measurements in different sand samples from “Las Canteras” beach taken in 2016 and 2018.

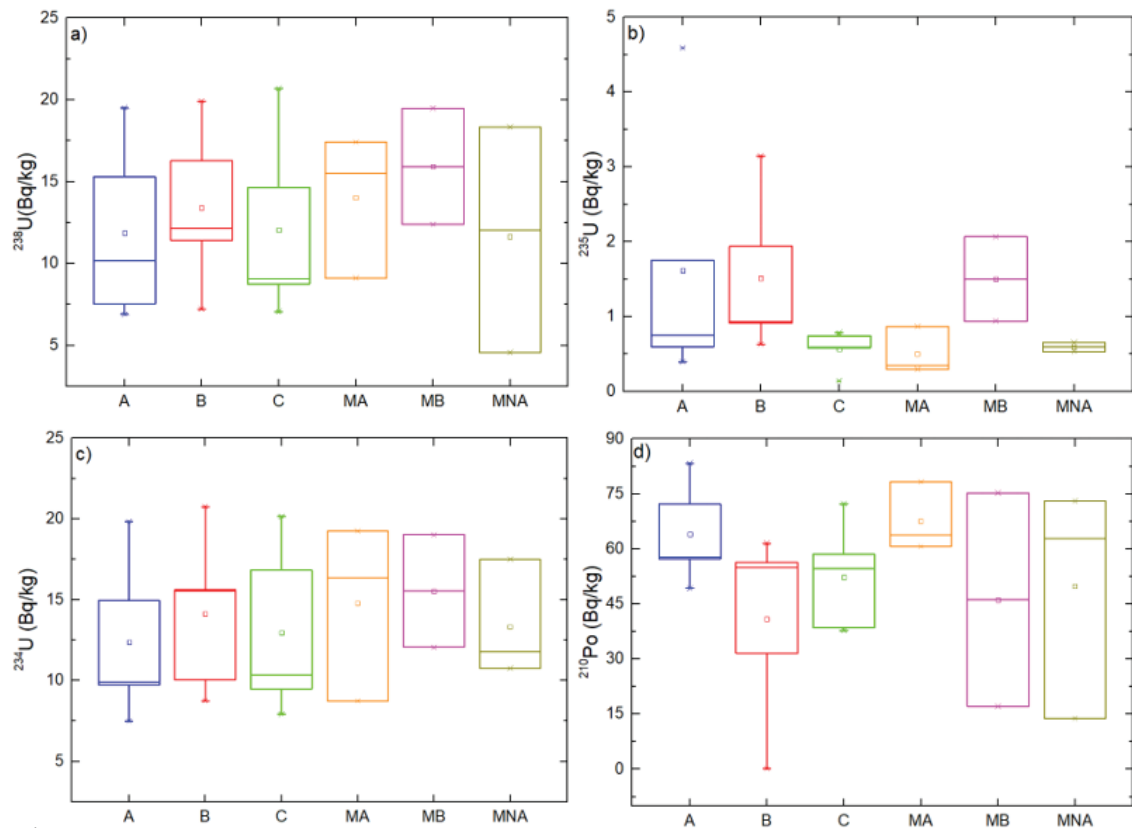


Figure 10.- Boxplot of the activity values of each radionuclide studied in sand samples from “Las Canteras” beach, obtained with different digestion methods. A means 24 h of digestion, B 48 h of digestion, C 72 h of digestion, MA corresponds to the first microwave digestion, MB to the second microwave digestion and MNA refers to microwave digestion with the second triplet of acids.

Figures 10a and 10c correspond to the activity concentration values of  $^{238}\text{U}$  and  $^{234}\text{U}$  respectively. It is shown that in both cases mean activity concentrations found for each digestion methods do not present great variation, finding mean activity values between 10 and 16 Bq/kg for both radionuclides. In Figure 10b the values of the mean activity concentration of  $^{235}\text{U}$  appear, fluctuating between 0.5 and 1.6 Bq/kg approximately. These activity concentrations are much smaller than those of  $^{234}\text{U}$  and  $^{238}\text{U}$  and present a higher dispersion. This occurs due to the small amount of  $^{235}\text{U}$  that can be found in environmental samples, in this case in sand samples. Figure 10d shows a great variability in the mean activity concentration of  $^{210}\text{Po}$ . The values ranged between 35 and 65 Bq/kg.

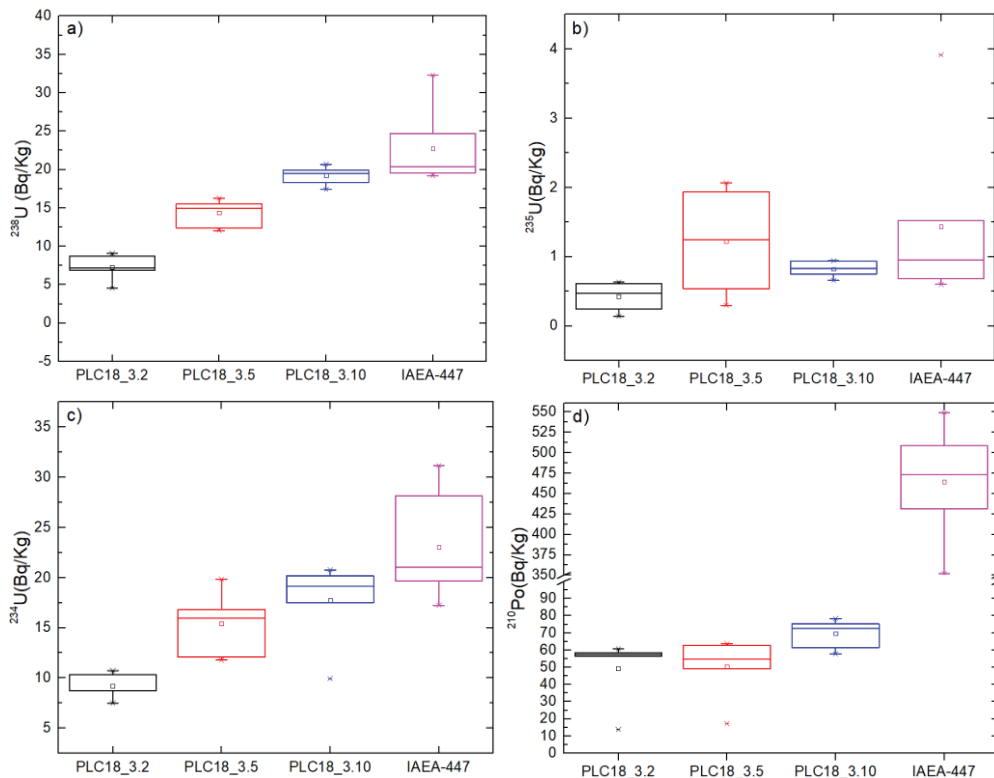


Figure 11.- Boxplot of the activity values of three sand samples of 2018 from “Las Canteras” beach and reference sample IAEA-447 of  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$ ,  $^{210}\text{Po}$ , obtained with all digestion methods.

On the other hand, Figure 11 shows the values of the activities concentrations of the radionuclides  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$ ,  $^{210}\text{Po}$  for three sand samples and reference sample IAEA-447, representing the results obtained by all digestion methods together for each sample. At first sight, the dispersion of the data in most of the sand samples for the four radionuclides is quite low, meaning that the digestion methods would not influence much in the results. However, the sample PLC18\_3.5 and reference sample IAEA-447 present a greater dispersion that could indicate the influence of some digestion method.

### 3.2 Activity concentrations of $^{238}\text{U}$ , $^{235}\text{U}$ , $^{234}\text{U}$ and $^{210}\text{Po}$ in referenced samples

The reference material IAEA-448 did not show good results when measuring the activity concentration of  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$ . The explanation to this is related to the fact that this reference sample has very high  $^{226}\text{Ra}$  content and the peaks of  $^{226}\text{Ra}$  and Uranium in the spectrum overlap. In addition, this reference sample is a contaminated bulk soil from an oil field. This means that the results in activity concentrations mainly related to the external contamination of the soil and not to the activity of the radionuclides of the materials that compose the soil itself. Therefore, this soil has no similarity with the behaviour of the sands, whose activities come from the materials that compose their sediments. Otherwise, the standard sample IAEA-RGU-1,

which is a preparation of dilution of a uranium ore, had very high values of activity concentration and makes it difficult to compare it with the sand samples. For this reason, it was finally decided not to use this two reference samples for later comparison.

Table 9 shows the activity concentration values of  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$  for the reference material IAEA-447. The results resemble the certified values presented in Table 2. In addition, these values are of the order of the values obtained for the sand samples from Las Canteras beach, being the  $^{210}\text{Po}$  slightly higher. Therefore, having a similar behaviour as the sand samples, this pattern sample was used for the rest of the analysis.

| <i>IAEA-447</i> | $^{238}\text{U}$ | $^{235}\text{U}$ | $^{234}\text{U}$ | $^{210}\text{Po}$ |
|-----------------|------------------|------------------|------------------|-------------------|
| A               | 19.17±1.20       | 0.60±0.15        | 19.65±1.22       | 352.19±13.99      |
| B               | 20.52±1.21       | 0.68±0.15        | 21.50±1.25       | 431.46±17.49      |
| C               | 20.29±1.36       | 0.77±0.20        | 20.58±1.38       | 445.17±17.66      |
| MA              | 19.54±1.00       | 1.14±0.17        | 17.24±0.91       | 500.41±18.93      |
| MB              | 32.30±1.50       | 1.52±0.20        | 31.18±1.46       | 508.27±18.83      |
| MNA             | 24.68±1.355      | 3.916±0.41       | 28.17±1.499      | 548.81±21.75      |

Table 9.- Activity concentration (Bq/kg) values for the radionuclides of the reference material IAEA-447.

### 3.3 Proficiency test results

From activity concentrations obtained by alpha spectrometry, the statistical parameters aforementioned in section 2 were calculated: RB (Eq. 3),  $U_{\text{test}}$  (Eq. 4) and  $P_{\text{Score}}$  (Eq. 5) according to IAEA (2011).

On the one hand, certified values of IAEA-447 (Table 2) have been used to calculate the statistical parameters corresponding to the results obtained by the different digestion methods in the laboratory. On the other hand, since it was not available a reference material with certified activity values for sands, the activity of precursor radionuclides measured by gamma spectrometry of the same sands samples measured in this work were used as reference values for the proficiency test. This can be done due to the secular equilibrium of the precursor radionuclides and its daughters measured by alpha spectrometry. The secular equilibrium occurs between the members of the radioactive decay series, due to the very low disintegration constants (long half-lives) of the precursor radionuclides of each series. This means that in geochemically closed systems, after a time determined by the half-life of the precursor radionuclide, the activity of all the isotopes in the decay series is equal to that of the radionuclide that originates them (Enresa, 1994). Hence, for  $^{238}\text{U}$  and  $^{234}\text{U}$ , values of  $^{226}\text{Ra}$  (precursor) were taken and for  $^{210}\text{Po}$  the values of  $^{210}\text{Pb}$  (precursor) were selected, according to the natural radioactive decay series (Annex II). In the case of  $^{210}\text{Po}$  it should be noted that  $^{210}\text{Po}$  is a natural radionuclide that has a half-life of 138 days. This means that in samples with more than 138 days (sand samples from 2016), all the  $^{210}\text{Po}$  will be in secular equilibrium with  $^{210}\text{Pb}$ . However, in the newer samples (sand samples from

2018) this equilibrium will not occur, since they had less than 138 days, and they will contain  $^{210}\text{Po}$  in excess. Moreover, the uncertainty for the gamma spectrometry measurement of  $^{210}\text{Pb}$  is high so the comparison with this radionuclide might not be completely correct. Noteworthy is the particularity of  $^{235}\text{U}$ . The reference activity concentrations of this radionuclide used for sand samples were those of the gamma emission line in 186 keV. In a sample containing both  $^{226}\text{Ra}$  and  $^{235}\text{U}$ , according to Ebaid et al., (2005), the  $^{226}\text{Ra}$  should contribute with approximately 58.3% of the total count of the peak rate in the 186 keV region, while  $^{235}\text{U}$  would contribute approximately with 41.7%. This percentage of  $^{235}\text{U}$  is calculated by the following expression:

$$A_{^{235}\text{U}} = \frac{0.424 \times A_{186} \times 0.0359}{0.572} = A_{186} \times 0.0266 \quad (6)$$

where  $A_{186}$  is the activity concentration of  $^{226}\text{Ra}$  in line 186 keV region of gamma emission. As the sample contained both  $^{226}\text{Ra}$  and  $^{235}\text{U}$  the activity concentrations of  $^{235}\text{U}$  measured by gamma spectrometry were calculated following Eq. (6) and were compared with the values of this radionuclide measured by alpha spectroscopy.

For evaluating which was the most optimal digestion method the two proficiency tests described on the previous section (Figure 9a and 9b) were used. First, the tests were applied to the reference sample IAEA-447. The results are shown in Table 10.

| IAEA-447 | (Shakhashiro et al., 2012) |                  |                  |                   | (Osvath et al., 2016) |                  |                  |                   |
|----------|----------------------------|------------------|------------------|-------------------|-----------------------|------------------|------------------|-------------------|
|          | $^{238}\text{U}$           | $^{235}\text{U}$ | $^{234}\text{U}$ | $^{210}\text{Po}$ | $^{238}\text{U}$      | $^{235}\text{U}$ | $^{234}\text{U}$ | $^{210}\text{Po}$ |
| A        | 4.11                       | 2.10             | 2.68             | 1.46              | -16.74                | -13.67           | -41.37           | -9.85             |
| B        | 0.42                       | 1.15             | 2.17             | 0.20              | 2.00                  | -7.55            | -33.24           | -1.39             |
| C        | 1.09                       | 1.20             | 1.24             | 0.76              | 5.24                  | -8.61            | -24.41           | -5.61             |
| MA       | 3.61                       | 1.57             | 0.65             | 3.74              | 29.74                 | 11.17            | 11.32            | 29.20             |
| MB       | 5.25                       | 5.94             | 2.41             | 5.64              | 20.16                 | 45.50            | 48.92            | 43.03             |
| MNA      | 3.61                       | 2.07             | 7.09             | 3.75              | 18.30                 | -11.98           | 289.92           | -20.90            |

Table 10.- Results of the proficiency test according to Shakhashiro et al., (2012) and Osvath et al., (2016) for reference IAEA-447 sample. A: digestion in oven for 24 hours. B: digestion in oven for 48 hours. C: digestion in oven for 72 hours. MA: aliquot A from the microwave digestion. MB: aliquot B from the microwave digestion. MNA: microwave digestion with another triplet of acids. Accepted in green, accepted with warning in orange and not accepted in red.

The results given by both proficiency test show that the digestion method B and the digestion method C are the ones with the largest accepted measurements, with all the radionuclides accepted for both methods and in both tests. Nevertheless, it can also be said that in general, a high number of accepted measurements are obtained with all methods. These results can give an idea of how the different digestion methods work in a reference sample with stipulated values of activity concentration.

Figure 12 and Figure 13 show the results obtained for the two proficiency tests when analysing the results obtained for sand samples and the reference sample IAEA-447 together by each digestion method. The results are given in terms of accepted, accepted with danger (warning), not accepted and without data.

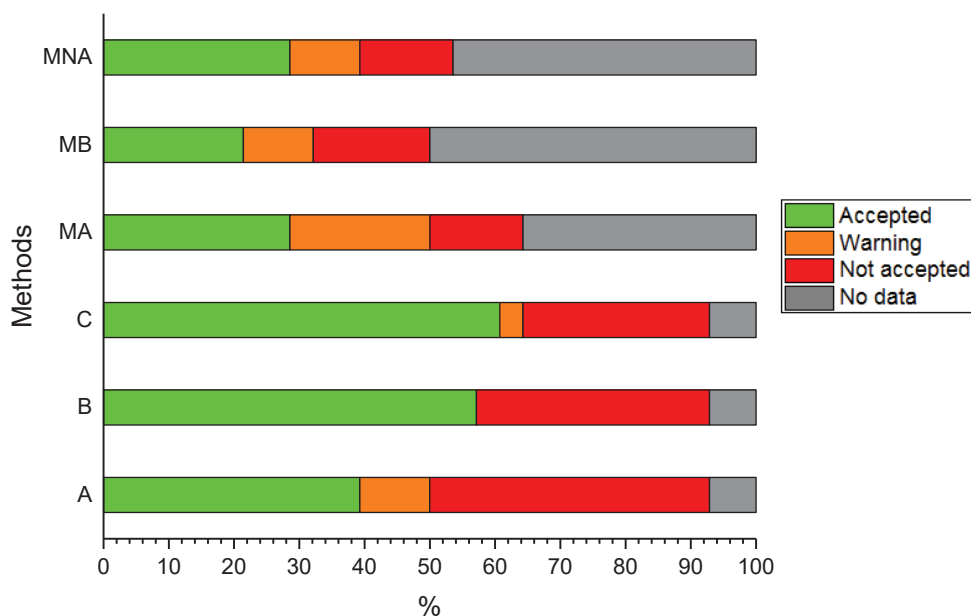


Figure 12.- Proficiency test according to Shakhashiro et al., (2012). A: digestion in oven for 24 hours. B: digestion in oven for 48 hours. C: digestion in oven for 72 hours. MA: aliquot A from the microwave digestion. MB: aliquot B from the microwave digestion. MNA: microwave digestion with another triplet of acids.

The results obtained for the test of Shakhashiro et al., (2012) and represented in Figure 12 show that the results for method A were 39% of accepted samples, 11% of samples warning and 43% of samples not accepted from the total. For the method B, the total of accepted samples corresponds to 57% and the samples not accepted to 36%. For the method C, 61% of accepted samples were obtained, 4% of warning and 29% of not accepted. Regarding microwave digestion, the results obtained for MA and MB have respectively a percentage of accepted measurements of 29% and 21%, warning 21% and 11% and not accepted 14% and 18%. Finally, observing the results in the MNA method, the accepted measurements are a total of 29%, 11% warning and 14% not accepted.

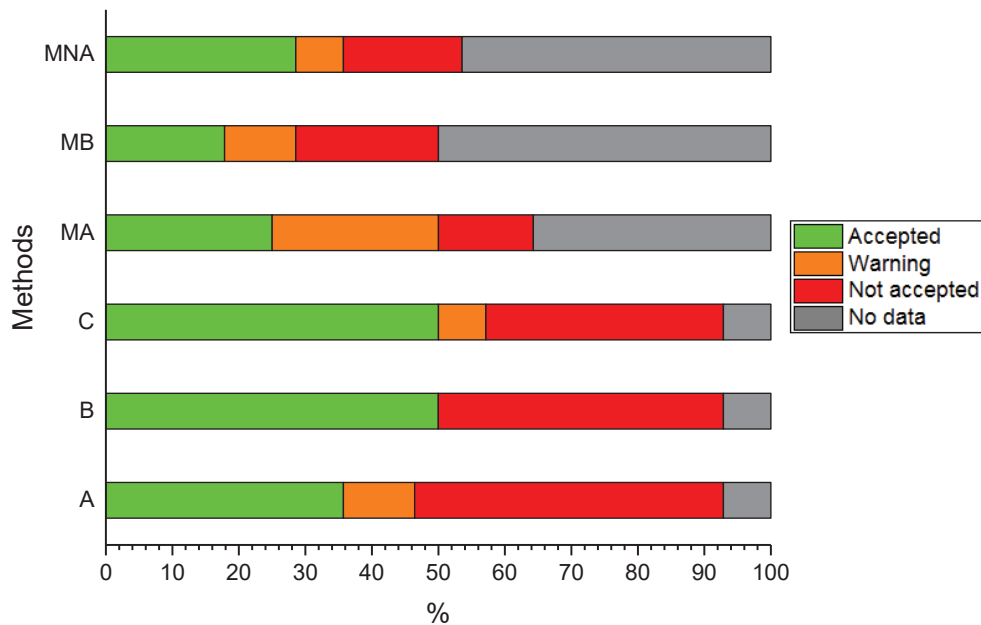


Figure 13.- Proficiency test according to Osvath et al., (2016). A: digestion in oven for 24 hours. B: digestion in oven for 48 hours. C: digestion in oven for 72 hours. MA: aliquot A from the microwave digestion. MB: aliquot B from the microwave digestion. MNA: microwave digestion with another triplet of acids.

Following the test of Osvath et al., (2016), a more restrictive method the results obtained vary regarding the previous ones (Figure 13). The results obtained for the method A were a 36% of samples accepted, a 11% of warning and a 46% of samples not accepted from the total. For the method B, the total of accepted samples corresponds to 50% and the samples not accepted to 43%. For the method C, a 50% of accepted samples was obtained, 7% of warning and 36% of not accepted. In the case of the microwave digestion, the results obtained for MA and MB have respectively a percentage of accepted samples of 25% and 18%, an amount of warning of 25% and 11% and not accepted samples of 14% and 21%. Finally, observing the results for the MNA method, the accepted ones were a total of 29%, a 7% was obtained for warning and a 18% of not accepted samples.

In summary, the results given by the two proficiency tests suggest that the method with the highest number of accepted samples is the method C, followed by method B. This implies that apparently the most optimal digestion method would be the oven digestion during 48 to 72 hours. However, it should be mentioned that the microwave digestion method used (MA and MB) followed the protocol given for soils by EPA, (2007). Nonetheless, this protocol is stipulated for soils in general not sand in particular so some variation in the pressure or temperature could be necessary for a better result. Besides, when changing the acid triplet, a small improvement appears in the percentage of accepted samples. Nevertheless, if the warning and not accepted percentages are taken into account, there is really no improvement in this digestions

method. Also, it is important to highlight that fewer data were obtained from the microwave digestions (MA, MB and MNA). Hence, it should be necessary to make more microwave digestions including the variation aforementioned, in order to rule out these methods against the oven digestion.

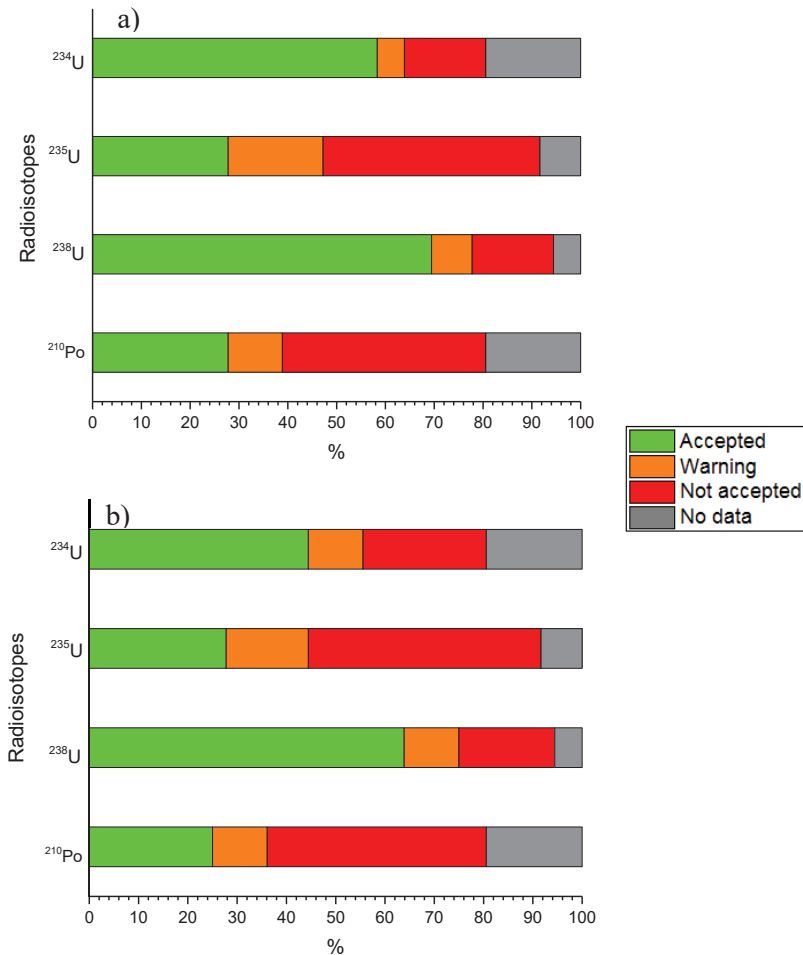


Figure 14.- Proficiency test for radionuclides according to (a) Shkhashiro et al., (2012) and (b) Osvath et al., (2016)

Figure 14 shows the results of both proficiency tests, but in this case for each of the radionuclides using the results of all digestion methods. The number of not accepted measurements for  $^{210}\text{Po}$  and  $^{235}\text{U}$  it was quite high. As it has previously been  $^{210}\text{Po}$  in the most recent samples is in excess, so the equilibrium secular with  $^{210}\text{Pb}$  does not occur and the comparisons probably was not correct. However, the comparison with the reference sample IAEA-447 was relatively well (Table 10). Thus, these results suggest that the problems with  $^{210}\text{Po}$  determination could not be related with of the digestion method used, but a mistake of the comparisons with the sands of less than 138 days (all samples of the campaign of 2018). For  $^{235}\text{U}$ , the problem probably is related with the fact that the values of this radionuclide are so small since there is so little in nature. Therefore, it is very complicated to determine, and thus, the error might be again on the comparison with gamma measured  $^{235}\text{U}$ .



Finally, for the radionuclides  $^{234}\text{U}$  and  $^{238}\text{U}$  have the higher accepted samples, being  $^{238}\text{U}$  the one with more accepted. Hence, it could be said that the  $^{226}\text{Ra}$  comparisons are correct and the adequate digestion methods were used. As the  $^{238}\text{U}$  is the radionuclide with the highest number of accepted samples in the set of all digestion methods, the remaining radionuclides,  $^{210}\text{Po}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$ , were represented in front of  $^{238}\text{U}$  in Figure 15. The purple square on the scatter plots limits the RB value in which the measurements are accepted according to the Table 8.

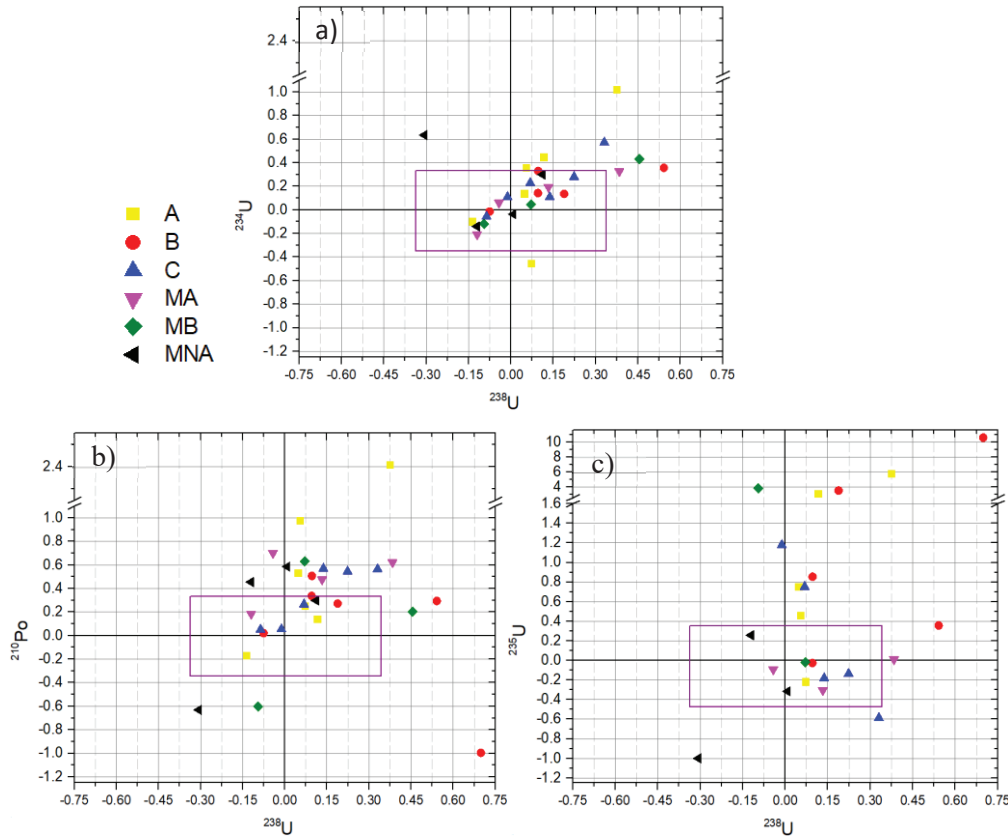


Figure 15.- Scatter plots of Relative Bias of  $^{210}\text{Po}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  represented in front of  $^{238}\text{U}$

Figure 15a shows a data dispersion relatively low, corroborating the information provided in Figure 14, which showed that all digestion methods were suitable for extracting  $^{234}\text{U}$  and  $^{238}\text{U}$ . However, for  $^{210}\text{Po}$  and  $^{235}\text{U}$  the dispersion found is quite high (Figures 15b and 15c). In the case of  $^{210}\text{Po}$ , there is an overestimation of the data that might be related with the polonium in excess of the newer samples. In the case of  $^{235}\text{U}$ , as it has been mentioned before, the high dispersion of data could be related with the low concentration of this radionuclide in the samples.

Considering all these results and since all the radionuclides are going to be extracted at the same time, if one had to choose a more optimal digestion method for it, the method C would be chosen. This is because this method is one that obtained better results when applying the proficiency test.

## 4. CONCLUSIONS

The aim of this work was to analyse the influence of the digestion method in alpha spectrometry measurements of uranium and polonium radioisotopes in sand samples from Las Canteras beach (Gran Canaria). The main conclusions obtained from the results were:

1.- The  $^{238}\text{U}$  and  $^{234}\text{U}$  activity concentrations obtained for sand samples showed low variation respect to performed digestion methods. The  $^{210}\text{Po}$  presented a slightly higher variability of obtained activities and the highest variability was found for the  $^{235}\text{U}$ . On the other hand, when analysing the activity concentration values obtained for  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$  in the different samples it can be appreciated that samples PLC18\_3.5 and reference sample IAEA-447 present a greater dispersion in the data that could be related to the different digestion methods used.

2.- When analysing the activity concentration of  $^{238}\text{U}$ ,  $^{235}\text{U}$ ,  $^{234}\text{U}$  and  $^{210}\text{Po}$  for reference samples, the only reference sample that showed a similar behaviour to the sand samples was the IAEA-447. Thus, the reference samples IAEA-448 and IAEA-RGU-1 were not used for the rest of the analysis.

3.- For evaluating the influence of digestion method two proficiency tests by ALMERA were carried out. The main conclusions obtained from this analysis were:

1. When applying both tests in the reference sample IAEA-447, the results showed that, in general, there were a high number of accepted results. However, the most optimal methods were the method B (48 h digestion) and the method C (72 h digestion).
2. When analysing the results obtained in both tests for all the samples, sand and reference samples, it appeared that the method with more accepted measurements were the method C (72h digestion), followed by method B (48h digestion). However, it should be mentioned that in the case of microwave digestion the number of data evaluated was less than the number of data from oven digestion Moreover, in the case of the microwave digestion there are other factors that contribute to the optimization of the method, such as the variations in temperature and pressure. Hence, more microwave digestions should be done to completely rule out this method against the oven digestion.
3. The results of the proficiency tests analysed by radionuclides show that for the  $^{238}\text{U}$  and  $^{234}\text{U}$  determination all methods were suitable. However, the results obtained for  $^{210}\text{Po}$  indicated that there is an overestimation of the activity concentration values that could be related with the  $^{210}\text{Po}$  that decay in the oldest samples. In the case of  $^{235}\text{U}$  the results were not good with a high dispersion in

the data. This might be due to low amount of this element that can be found in each sample and the consequent difficulty when measuring this element.

Finally, if a digestion method should be selected, taking into account the results obtained in this study, the recommended method would be the oven digestion between 48 and 72 hours.

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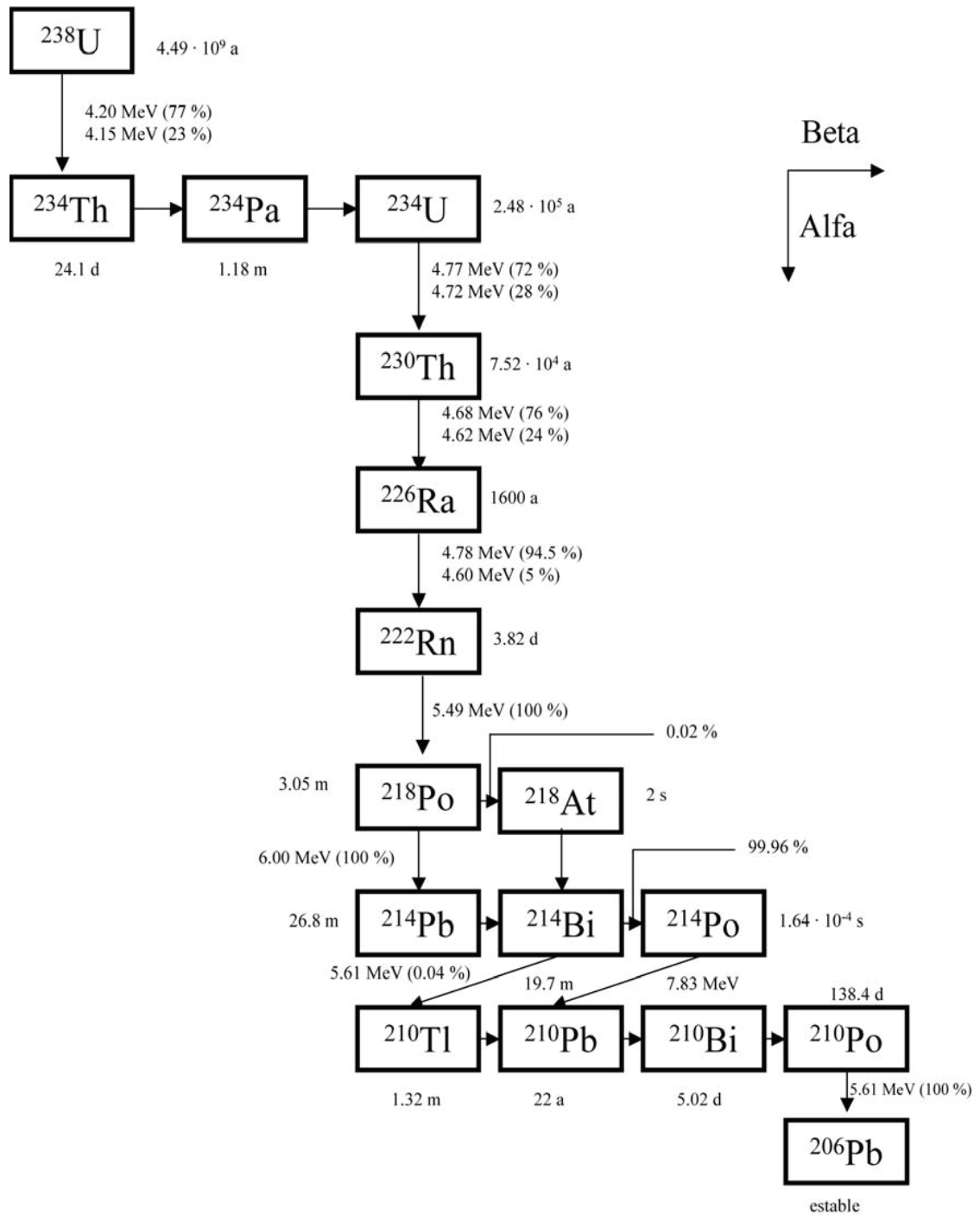
## ANNEX I

Table of activity concentration values (Bq/Kg) obtains in laboratory for the radionuclides of the sand samples from “Las Canteras” beach, taken in 2016 and 2018.

| <i>Samples</i> | <i><sup>238</sup>U</i> | <i><sup>235</sup>U</i> | <i><sup>234</sup>U</i> | <i><sup>210</sup>Po</i> |
|----------------|------------------------|------------------------|------------------------|-------------------------|
| PLC16_5.2A     | 7.5±1.3                | 0.40±0.28              | 9.7±1.5                | 72.3±4.3                |
| PLC16_5.2B     | 12.1±1.3               | 3.15±0.62              | 15.6±1.6               | 2.94±2.1                |
| PLC16_5.2C     | 7.1±0.6                | 0.59±0.15              | 7.9±0.7                | 38.5±2.2                |
| PLC16_5.3A     | 10.2±0.8               | 4.59±0.28              | 14.97±1.3              | 83.3±3.9                |
| PLC16_5.3B     | 11.4±1.6               | 0.91±0.46              | 10.1±1.7               | 31.7±2.1                |
| PLC16_5.3C     | 9.1±1.0                | 0.58±0.22              | 9.5±1.0                | 37.8±2.3                |
| PLC18_3.2A     | 6.9±0.8                | 0.60±0.20              | 7.5±0.8                | 57.3±2.8                |
| PLC18_3.2B     | 7.2±0.9                | 0.63±0.24              | 8.8±1.0                | 56.3±2.8                |
| PLC18_3.2C     | 8.8±0.8                | 0.14±0.08              | 10.4±0.8               | 58.5±2.9                |
| PLC18_3.2MA    | 9.1±0.6                | 0.34±0.11              | 8.7±0.6                | 60.7±2.7                |
| PLC18_3.2MNA   | 4.6±1.3                | -                      | 10.8±2.0               | 13.8±0.8                |
| PLC18_3.5A     | 15.3±1.2               | 1.75±0.33              | 19.8±1.4               | 49.2±2.4                |
| PLC18_3.5B     | 16.3±1.0               | 1.94±0.26              | 15.56±1.0              | 54.9±2.6                |
| PLC18_3.5C     | 14.6±0.9               | 0.75±0.14              | 16.8±0.9               | 54.7±2.7                |
| PLC18_3.5MA    | 15.6±0.9               | 0.30±0.09              | 16.4±0.9               | 63.8±2.8                |
| PLC18_3.5MB    | 12.4±0.9               | 2.07±0.30              | 12.1±0.9               | 17.1±0.7                |
| PLC18_3.5MNA   | 12.0±0.9               | 0.54±0.16              | 11.8±0.9               | 62.9±2.7                |
| PLC18_3.10A    | 19.5±1.2               | 0.75±0.16              | 9.9±0.7                | 57.8±2.7                |
| PLC18_3.10B    | 19.9±1.2               | 0.93±0.18              | 20.8±1.2               | 61.6±2.9                |
| PLC18_3.10C    | 20.7±1.2               | 0.78±0.16              | 20.2±1.2               | 72.3±3.4                |
| PLC18_3.10MA   | 17.4±1.0               | 0.87±0.16              | 19.3±1.1               | 78.3±3.4                |
| PLC18_3.10MB   | 19.5±1.0               | 0.94±0.16              | 19.0±1.0               | 75.2±3.3                |
| PLC18_3.10MNA  | 18.3±1.1               | 0.66±0.15              | 17.5±1.0               | 73.1±4.0                |

## ANNEX II

Natural radioactive series of  $^{238}\text{U}$ .





## **VALORACIÓN PERSONAL (PERSONAL ASSESSMENT)**

### *1.- Actividades desarrolladas durante la realización del TFT*

Durante la realización del Trabajo de Fin de Título se desarrollaron distintos tipos de trabajos, algunos de los cuales estaban incluidos en las prácticas externas. Por un lado, se realizó la digestión y extracción de uranio y polonio de patrones certificados (IAEA-447, IAEA-448, IAEA-RGU-1), de muestras de arena de la playa de las Canteras (PLC16\_5.2, PLC16\_5.3, PLC18\_3.2, PLC18\_3.5, PLC18\_3.10) recogidas algunas en una campaña en la que participé en las prácticas. (PLC18\_XX). Esto se llevó a cabo tal y como se describe en el capítulo 2 de este documento, y posteriormente se midieron por espectrometría alfa. Finalmente se realizó una búsqueda bibliográfica sobre el tema a tratar en el TFT y paralelamente a la realización de los experimentos se realizó la redacción del mismo.

### *2.- Formación recibida*

En el Máster de Oceanografía que tuvo lugar en el segundo cuatrimestre del curso 2017-18, se impartió la asignatura Oceanografía a Gran Escala y Mesoscala, donde se dio el tema Radioactividad Marina, y asistí a las sesiones en las que se impartió este tema. También fui a las tres sesiones de prácticas planificadas. Por último, cabe destacar toda la información, y todo tipo de bibliografía necesaria para comprender el estudio que iba a llevar a cabo (tanto tesis doctorales como artículos científicos), además de las explicaciones de mis tutores sobre cada uno de los temas, que me ayudaron a la hora de realizar las practicas externas, así como a la hora de realizar el trabajo de fin de grado.

### *3.- Nivel de integración e implicación dentro del departamento y relación con el personal*

En general las relaciones con el personal del departamento han sido cordiales y agradables, sin ningún incidente. Hay miembros del departamento no he llegado a conocer, si bien los que he conocido han sido todos amables y con predisposición a aclararme cualquier duda que me pudiera surgir. Cabe destacar a Ana del Carmen por su gran trabajo como mentora, ya que me ha ayudado en todo lo que en su mano estaba y su compañía en el laboratorio durante las largas horas de espera ha hecho la situación amena y agradable. También mencionar a mis tutores, Alicia Tejera Cruz y Pablo Martel Escobar, quienes a pesar de estar muy ocupados me han guiado sin problema durante las distintas fases del periodo de prácticas.

### *4.- Aspectos positivos y negativos más significativos relacionados con el desarrollo del TFT*



El aspecto positivo que cabe destacar es el hecho de que en cualquier momento cualquier duda se me solucionaba y tenía toda la información disponible para realizar el TFT .Y como aspecto negativo, el hecho de ser el TFT en inglés ha ralentizado el tiempo de realización, No se nos prepara desde el comienzo de la carrera para esto, y que la parte más importante de la carrera se tenga que tratar en una lengua que no se domina del todo es un impedimento muy grande.

#### *5.- Valoración personal del aprendizaje conseguido a lo largo del TFT*

Una vez realizado el TFT puedo decir que mi valoración personal es positiva. Durante la realización del TFT he aumentado mi nivel de conocimientos adquiridos en las prácticas sobre la Radioactividad Ambiental, interpretación de los resultados obtenidos, así como del dominio del inglés en temas de redacción y lectura. He aprendido a trabajar de manera autónoma y eficiente siempre apoyada por mis tutores.

En general estoy satisfecha del trabajo realizado, de mi crecimiento en este ámbito, a pesar de que aún me quede mucho por aprender dentro del tema de la Radiactividad Ambiental