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IAEA-TECDOC-1953

## **A Guide to the Use of Sediments in Reconstructing the Pollution History of Coastal Areas**



**IAEA**

International Atomic Energy Agency

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A GUIDE TO THE USE OF SEDIMENTS IN  
RECONSTRUCTING THE POLLUTION HISTORY  
OF COASTAL AREAS

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INTERNATIONAL ATOMIC ENERGY AGENCY

VIENNA, 2024

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## FOREWORD

From 2007 to 2012, the IAEA ran a regional project on the ‘Use of Nuclear Techniques to Address the Management Problems of Coastal Zones in the Caribbean Region’, RLA/7/012. The main purpose of the project was to develop and build capacity in the use of nuclear and isotopic methods in order to understand and propose strategies for reducing the degradation of coastal ecosystems and support integrated coastal zone management in the Greater Caribbean region. The project involved Colombia, Costa Rica, Cuba, Dominican Republic, Guatemala, Haiti, Honduras, Jamaica, Mexico, Nicaragua, Panama and the Bolivarian Republic of Venezuela, with support from France and Spain, and in collaboration with the United Nations Environment Programme.

One of the project’s main methodologies involved the use of  $^{210}\text{Pb}$  sediment dating to document environmental changes, including pollution. An important part of the project strategy was to produce a practical guide to sampling and preparing sediment cores for a historical reconstruction of pollution in the region’s coastal areas. It would be a guide to the main techniques to be used, planning the work and coordinating the activities to be conducted in the laboratories participating in the project.

To address this need, a group of experts in sediment pollution and dating came together in May 2007 for a regional workshop in Cumaná, Bolivarian Republic of Venezuela, to discuss the most appropriate strategies for the region. They drafted the first version of the guide, which was then discussed and revised in September 2007 during a regional course held in Cienfuegos, Cuba. From the outset, the guide was designed to be a living document that could be adapted to any needs or challenges encountered during the project’s implementation. This publication is the 2020 updated version of the document created in 2007. It incorporates the best practices that were identified, based on the experience acquired and results obtained over the course of the project, while preserving the validity of the methodologies and procedures described herein. While some of the specific information in this publication will not be directly applicable to other regions, the structure and approach might well be applicable and it can easily be adapted to other national or regional projects.

This guide is a product of project RLA/7/012. It was developed by and for scientists in the region as a guide to the use of sediments to assess current and historical levels of pollution in coastal areas (over roughly the last hundred years) by studying sediment cores dated using the radionuclides  $^{210}\text{Pb}$  and  $^{137}\text{Cs}$ . There is a special focus on sampling methods that provide a basis for dating, including an analysis of geochemical and sedimentological parameters, which help to improve the understanding of variation in rates of sediment accumulation and pollutant concentrations at study sites. Care was taken to ensure the quality of the analytical data on concentrations of pollutants, and the guide sets out the most common methods of normalizing these to assess pollution levels. This guide complements the book *Radiocronología de sedimentos costeros utilizando  $^{210}\text{Pb}$ : modelos, validación y aplicaciones* [Coastal sediment radiochronology using  $^{210}\text{Pb}$ : models, validation and applications] published by the IAEA in 2012, which was also a product of project RLA/7/012.

The IAEA thanks the Instituto de Oceanografía at the Universidad de Oriente (Bolivarian Republic of Venezuela) and the Centro de Estudios Ambientales de Cienfuegos (Cuba) for organizing the regional meetings that made possible the design and development of this publication, along with the experts and revisers who contributed to it and who are listed at the end.





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# 1. INTRODUCTION

## 1.1. BACKGROUND

Sediment is the accumulation of various types of material arising both from continental transport and biogenesis in situ. As they contain organic matter and clay, sediments can be excellent at transporting and accumulating both naturally occurring and anthropogenic substances such as heavy metals or persistent organic compounds.

In areas where the accumulated sediment has not undergone bioturbation, physical mixing or erosion, the sedimentary column is a comprehensive record of changes in bodies of water over time. It contains valuable historical information on the characteristics of the environment at the time it was formed and any changes over time resulting from anthropogenic activities [1]. The sedimentary column is therefore a very useful natural repository of data for reconstructing past environmental events. Such records can provide specific data on anthropogenic pollutant flows arising from urban and industrial development [2, 3].

In the absence of information on the state and evolution of pollution in the coastal zone, and if undisturbed sedimentary records can be obtained, studying the sedimentary column can make it possible to reconstruct the history of pollution from metals and metalloids (e.g. As, Cd, Co, Cr, Cu, Hg, Ni, Pb, V and Zn, commonly referred to as heavy metals) and persistent organic compounds (e.g. polycyclic aromatic hydrocarbons — PAHs — and pesticides). From these records, specific data can be obtained on changes in sediment accumulation rates and pollutant flows over time but a correct interpretation of this valuable information depends fundamentally on being able to establish a reliable accumulation rate.

The naturally occurring radionuclide  $^{210}\text{Pb}$  ( $T_{1/2} = 22.3$  years) is used to date sediments deposited in the preceding 100–150 years [4]. This method is also commonly used with  $^{137}\text{Cs}$ , an anthropogenic radionuclide whose presence worldwide in sediments is a result of the nuclear testing that was conducted around the world, mostly in the late 1950s and early 1960s.

A retrospective study of the pollution of aquatic systems using  $^{210}\text{Pb}$  sediment dating requires a suitable selection of sampling sites and samples that best preserve the original characteristics of the sedimentary column at the study site, i.e. the procedures used should avoid mixing, compacting or contaminating the sediments when collecting or processing them for later analysis.

This guide describes the basic processes involved in collecting and handling sediment cores for dating purposes, and includes a series of analyses of essential variables (e.g. sediment moisture and porosity) and complementary variables (e.g. grain size and organic and inorganic carbon content) for interpreting changes over time in both sediment accumulation rates and pollutant concentrations at study sites. It also recommends practices to ensure the quality of the analytical data and explains the normalization methods regularly used to assess levels of heavy metal pollution.

## 1.2. OVERALL OBJECTIVE

The overall objective is to reconstruct the history of pollution from metals and metalloids (As, Cd, Co, Cr, Cu, Hg, Ni, Pb, V and Zn) and persistent organic compounds (PAHs and pesticides) by studying  $^{210}\text{Pb}$ -dated sediment cores collected in the Greater Caribbean coastal zone.

## 1.3. SCOPE

This guide was developed by experts in pollution and radiometric dating from the Greater Caribbean region. It makes available to the reader the theoretical knowledge and practical experience acquired by these experts as researchers and trainers over the course of their careers, but also specifically in the development of project RLA/7/012 for the Latin America region, which is why many of the procedures reflect the prevailing conditions at the study sites and laboratories in that region. Some of the techniques and processes described may not apply directly to other regions. However, the philosophy behind the division of labour and the structuring of the work described in this guide are useful and could easily be adapted to suit the needs of other national or regional projects.

## 1.4. STRUCTURE

This guide is divided into eight sections. Section 1 sets out the background, objective, scope and structure of the guide. Section 2 describes the sampling design, in line with the objectives of project RLA/7/012. Section 3 explains the process of collecting the surface sediment samples — a stage preceding the collection of the sediment cores and recommended for study sites where no basic information is available to help with choosing a suitable sampling site for a historical analysis of pollution. Section 4 contains instructions for collecting sediment cores. Section 5 provides details of the procedure for recording information about the site, the conditions under which samples are taken of surface sediments and sediment cores, and the specific characteristics of the latter, using forms designed for the sampling strategy. Section 6 sets out the process of coding the samples, which aids in the organization of the material collected and its distribution for subsequent analysis. Section 7 describes the criteria for selecting and handling the sediment cores that will undergo  $^{210}\text{Pb}$  dating, and section 8 presents the analyses of essential and complementary variables to be carried out for a retrospective analysis of the pollution at the study sites. The analytical methods are described in detail in the annexes, which are available as supplementary files and can be found on the web page for this guide at: [www.iaea.org/publications](http://www.iaea.org/publications).

## 2. SAMPLING DESIGN

### 2.1. GOALS

Sampling design is one of the most important phases in a research project. Much of the success of each study programme lies in clearly defining the goals and the strategy for sampling. In our particular case, the aim is to:

- Obtain undisturbed sediment cores that allow the recent geochronology to be reconstructed using the  $^{210}\text{Pb}$  and  $^{137}\text{Cs}$  methods;

- Determine the concentration of elements of environmental interest, such as Ag, As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Se, Sn, V and Zn, and those of geochemical interest (Al, Ba, Br, I, Ca, Cl, Fe, Mg, Mn, Na, K, P, Rb, Si, Sr, Ti and Zr) that have accumulated in the sedimentary column at the sites of interest;
- Identify and quantify the content of polycyclic aromatic hydrocarbons (PAHs) and organochlorine pesticides (OCPs) present in the sedimentary column in the different study areas. The specific compounds to be studied are:
  - PAHs: acenaphthene, acenaphthylene, anthracene, benzo[a]anthracene, benzo[a]pyrene, benzo[e]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene, biphenylene, chrysene, dibenzo[a,h]anthracene, dibenzothiophene, phenanthrene, fluoranthene, fluorene, indeno[1,2,3c,d]pyrene, naphthalene, perylene, pyrene, 1-methylnaphthalene, 2-methylnaphthalene, 2,6-dimethylnaphthalene, 2,3,5-trimethylnaphthalene, 1-methylphenanthrene;
  - OCPs: aldrin, alpha-chlordane, dieldrin, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, endrin, endosulfan I, endosulfan II, endosulfan sulphate, endrin aldehyde, endrin ketone, alpha-HCH, beta-HCH, delta-HCH, gamma-HCH (lindane), heptachlor, heptachlor epoxide, methoxychlor, gamma-chlordane, cis-nonachlor, trans-nonachlor, 2,4'-DDD, 2,4'-DDE, 2,4'-DDT;
- Determine, if possible, the degree of pollution due from metals, PAHs and OCPs at each study site;
- Quantify the changes over time in the flows ( $\text{g}\cdot\text{cm}^{-2}\cdot\text{a}^{-1}$ ) of the pollutants of interest in each of the sampling areas.

## 2.2. SELECTING SAMPLING SITES

### 2.2.1. Basic information for sampling

Once the area of interest has been decided upon, selecting the sediment core collection sites requires as much basic information as possible, including:

- Aerial/satellite photos;
- A bathymetric map;
- Dredging records;
- Local hydrodynamics;
- Inventory of pollution sources;
- Relevant catchment areas;
- Grain size distribution map;
- Surface pollution maps;
- Water quality data and information on benthic stocks.

### 2.2.2. Characteristics of the sampling site

When creating a historical reconstruction of pollution using sediment cores, the recommended sites for sediment core collection are those with:

- Preferential deposition of fine-grained (muddy) sediments;
- A sedimentation rate between 0.25 and 1.0 cm/a;

- A minimum depth of 15 m;
- A certain degree of compaction (avoid sediment that is too porous and susceptible to resuspension); and
- Hypoxia/anoxia conditions (dissolved oxygen content <0.1 mg/L) with minimal biological activity (benthic macroinvertebrates, bivalves, polychaetes, etc.).

To reduce the potential for problems with mixing, it is advisable to avoid sites associated with known dredging activity, trawling, anchorage of vessels or passage of vessels propelled by a pole pushed against the bottom. It is also recommended that sampling be carried out during the dry season.

Sites should be chosen that show signs of anthropogenic impact, such as industrial discharge or changes in land and water use. It is recommended to stay within the area affected by the sources of pollution (which will depend on the site).

For sites where no preliminary work has been done that would provide basic information for selecting a sampling site, it is recommended that a surface sediment survey be carried out in the area of interest before collecting sediment cores (see section 3).

### **2.2.3. General recommendations for conducting sampling**

The following are some basic recommendations for conducting a sampling campaign:

- Draw up a plan for the sampling campaign with everyone who will be involved;
- Check that everyone involved has the training needed to accomplish the objectives established in the sampling design;
- Comply with safety measures during sampling and navigation;
- Ensure that everyone involved in the sampling wears suitable gloves and avoids using sunscreen and repellents, as these products can contaminate the cores. Nitrile gloves are recommended, as some other materials may contaminate the samples and affect the analysis results for organic compounds;
- Do not smoke during the sampling;
- If possible, stop the vessel's engine and collect the sample from the opposite side to where ballast or bilge water is discharged.



### 3. SURFACE SEDIMENTS

The recommended steps for studying surface sediments are presented in Fig. 1. This sequence starts with the selection of the sampling site according to the instructions in section 2.2 above.

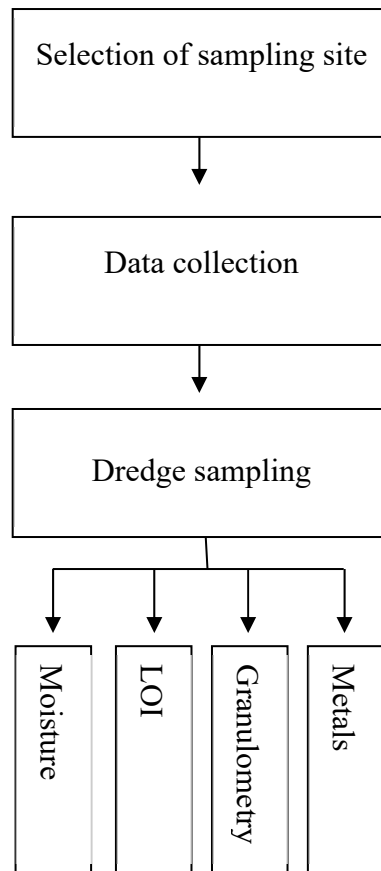


FIG. 1. Workflow for the study of surface sediments.

#### 3.1. SAMPLING

Surface sediments should be sampled using a grid of 20 to 30 sampling stations, using a Van Veen grab.

- The dredge must be washed before use to remove any traces of sediment from previous sampling and rinsed with water from the sampling site after each use;
- Plastic containers and utensils used to handle the samples for metal analysis should be washed first in HCl (2N) and then in HNO<sub>3</sub> (2N), then rinsed with distilled water and air-dried away from sources of contamination;
- Basic information on each sampling site should be recorded using a form designed for that purpose (see section 5);

- Care should be taken to collect only the surface layer of sediments collected by the dredge (1 cm thick) to ensure a consistent sampling strategy and avoid mixing recent sediment with any that is too old. For this separation process, a spatula made of a suitable material should be used in order to ensure that the sample does not become contaminated, e.g. plastic for samples being analysed for heavy metals;
- Sediment samples should be placed in tared plastic bags and the wet weight recorded;
- To avoid confusion, and material being lost during different steps of the study process, the samples should be carefully identified and labelled following the instructions given in section 6. The sample bag should be clearly labelled out the outside and then should ideally be placed in a second plastic bag to prevent the label from being rubbed off.

### 3.2. MOISTURE ANALYSIS AND LOSS ON IGNITION

Sediments should be analysed for moisture and loss on ignition (as described in detail in Annex I of the supplementary files to this guide). Both parameters are crucial to ensure the best conditions for reliable dating of the sediment cores. The moisture percentage allows the sediment's in situ density to be estimated, which is essential in evaluating the effect of depth on the sediment's compaction. The dry weights obtained during the moisture analysis are used to estimate the sediment's mass depth, a fundamental variable for  $^{210}\text{Pb}$  dating (see [5]). The analysis also enables an estimate of the sediment's organic matter content [6], the variation of which according to depth can provide clues about potential sediment disturbance [7].

For the moisture analysis (%) the samples should ideally be freeze dried, or else oven dried at a temperature below 45°C, and the dry weight recorded. This temperature was chosen to prevent the loss of volatile components such as mercury or organic compounds. There may be losses of methyl mercury or of more volatile organic compounds, but such losses may also have occurred before sample collection owing to the region's high temperatures.

The samples should be weighed using grain scales having a precision of 0.001 g or better. It is recommended to weigh the dry sample until the reading is constant, i.e. until the difference between each weighing is 1% or less. The sample's moisture content is calculated as a percentage using Eq. (1):

$$\text{Moisture (\%)} = \frac{(\text{Fresh weight} - \text{Dry weight})}{\text{Fresh weight}} \times 100 \quad (1)$$

Samples should be broken up by hand to separate the aliquots required for the analyses to be performed (which are described in section 8).

For the loss on ignition analysis (LOI<sub>550</sub>), 0.5 g of dry sediment is weighed in a porcelain crucible (using a grain scale having a precision of 0.001 g or better). The sample is calcined in a muffle furnace at 550°C for four hours (starting from the moment the desired temperature has been reached and has stabilized). The sample is allowed to cool to 100°C and is transferred to a desiccator, where it is left to cool down to room temperature before being reweighed. The LOI<sub>550</sub> is calculated using Eq. (2):

$$\text{LOI}_{550} (\%) = \frac{(\text{Initial weight} - \text{Calcined weight})}{\text{Initial weight}} \times 100 \quad (2)$$

The laboratories must ensure that the analysis of samples is reproducible, and it is therefore important to consider the various factors affecting LOI<sub>550</sub> analysis, such as where the samples are located in the muffle furnace. It is recommended to place them near the back of the furnace, since those near the door are subjected to lower temperatures than those at the back. They should also be well distributed in the crucibles, which should not be too small.

### 3.3. PREPARING SAMPLES FOR ANALYSIS

After the moisture and LOI<sub>550</sub> analyses have been performed, the rest of the sample is divided and put into plastic bags of a suitable size for the sample quantities required for the subsequent analyses to be performed (see section 8). Note that a minimum amount of material has been established for each analysis in order to ensure that samples with a high moisture content contain sufficient sediment for the respective analyses.

- Each bag should be coded following the instructions in section 6.
- 1 to 2 g of UNGROUND sediment is set aside for grain size analysis by laser diffraction.

The rest of the sample is ground in a mortar, preferably one made of agate. Before the mortar is used it must be washed using phosphate-free soap and rinsed under running water, then rinsed once with distilled water and once with diluted 2M HCl, and then finally rinsed three times with deionized water to remove all acid residues. It is recommended to clean the agate mortar thoroughly between samples.

## 4. SEDIMENT CORES

### 4.1. CORE SAMPLING STRATEGY

Once the documentary information generated by previous studies in the area has been compiled, the most suitable sites for collecting the sediment cores can be chosen. The sampling guidelines used in project RLA/7/012 are given below:

- Number and location of stations: three sampling stations (A, B and C) are chosen for each area of interest;
- Number of sediment cores: because requirements may vary for the handling of samples depending on the analyses to be performed (plastic containers for analysis of metals, glass or metal for analysis of organic compounds), three sediment cores are collected at each station;

- Purpose of the cores: as shown in Table 1, Core I is used for radionuclide analysis (geochronology), metals and geochemical parameters; Core II is used for organic pollutants (PAHs and OCPs) and Core III is saved as a control;
- For collecting the samples a UWITEC gravity corer is used, which allows three cores to be collected simultaneously, although it also allows cores to be collected individually if necessary;
- All information relating to sediment core sampling, including the sampling site's location and characteristics, should be carefully recorded on the sediment core collection form (see section 5).

TABLE 1. NUMERICAL CODES FOR CORES

Core	Analysis to be carried out
I	Radionuclides, metals and geochemical parameters
II	Organic pollutants
III	Control

## 4.2. CLEANING THE CORE SAMPLING EQUIPMENT

### 4.2.1. Sampling equipment

Corers are to be washed before use to remove any traces of sediment from previous sampling and rinsed each time with water from the sampling site.

### 4.2.2. Equipment for heavy metals

Before use, the plastic containers and utensils for handling samples for metal analysis should be washed first in HCl (2N) and then in HNO<sub>3</sub>(2N), then rinsed with distilled water (or Milli-Q) and air-dried away from sources of contamination.

### 4.2.3. Material for organic pollutants

Glass containers and utensils used for samples to be analysed for organic pollutants should be washed with soap and water before use.

It is recommended to heat glassware at 450°C for at least four hours to remove any traces of organic pollutants or, alternatively, to rinse glassware and Teflon with pesticide grade acetone to remove any water and then with hexane to remove organic compounds.

## 4.3. IN SITU PARAMETERS

It is recommended that the following additional parameters be determined during sample collection:

- Temperature (multiparameter probe);
- Salinity (multiparameter probe);
- Dissolved oxygen (multiparameter probe);
- Water column depth (echo sounder);
- Weather parameters (air temperature, wind speed and direction), if the vessel is equipped with a weather station.

#### 4.4. HANDLING CORES IN THE FIELD

##### 4.4.1. Inspecting the core

Given that the main objective is to reconstruct the recent geochronology of the pollution (in the last 100–150 years), each time a core is collected it should be inspected visually to verify:

- That it has a horizontal surface;
- That there are no traces of mixing or disturbance, either physical or biological, such as cracks, trapped air bubbles, resuspension, patches of mixed sediment of different colours or textures, bivalve shells and polychaete tubes, etc.

If a core has an inclined surface or shows any sign of mixing, it is recommended to discard it and try again.

##### 4.4.2. Important steps to follow during sampling

Once the sediment cores have been taken at the selected station, the following procedure is to be observed:

- Fill in the data for the cores in the sediment core collection form (section 5.2.);
- Code and label the cores carefully, as indicated in section 6.2;
- Take a technical photograph of the core using a metric ruler for scale. If this cannot be done in the field, it should be done in the laboratory as soon as is practicable;
- During sampling, the cores are to be kept in the coolest part of the vessel, protected from the sun and any risk of contamination by dust or rain (put a lid or plastic bag on each tube);
- Move the cores carefully and avoid knocking them;
- Handle them very carefully, making sure they remain in an upright position at all times and storing them in a rack to prevent any tilting that could cause mixing;
- Keep them refrigerated (at 4°C), wherever possible.

#### 4.5. HANDLING CORES IN THE LABORATORY

It is recommended that the cores be left to stand in the laboratory, at the location where they will be processed, for at least 24 hours to allow the particles at the core's watery surface to settle.

If a technical photograph was not already taken in the field, a photograph is taken of the core using a metric ruler for scale.

Remove as much supernatant water as possible using a flexible transparent plastic tube less than 1 cm in diameter, taking care not to resuspend or siphon off the surface sediment. The remaining supernatant water is extracted using a pipette, partially evaporated (maximum volume 10 ml) in an oven at less than 60°C, and then added to the first section of the core.

#### **4.5.1. Cutting the core**

The cores are to be cut using the stainless steel cutting tool provided with gravity corers (e.g. UWITEC™ or similar). Care should be taken to decontaminate the cutting tools between samples by rinsing them with distilled water. The samples for radionuclide and metal analysis are placed in plastic bags and coded according to section 6 of this guide.

To achieve good resolution in the chronological analysis, the cores should be cut into sections 1 cm thick. Given that the sedimentation rate in a coastal zone is 0.25 to 1.0 cm/a, and assuming an average rate of 0.5 cm/a, 50 cm of core would be expected to contain sediments accumulated over the course of 100 a. Given that the tube of the UWITEC™ corer has a diameter of ~9 cm, a layer 1 cm thick would contain approximately 95 g of wet sediment ( $20.25 \text{ cm}^2 \times 3.1416 \times 1 \text{ cm} \times 1.5 \text{ g/cm}^3$ ). As the layers closer to the surface often have high moisture levels (>80%), it is likely that there will not be enough material for additional analyses, so sample use should be prioritized on a case-by-case basis (e.g. reusing samples that were analysed using non-destructive techniques, combining sections, etc.).

It must be kept in mind that extruding the sediment core causes friction between the sediment and the inner surface of the sampling tube and that, given the stickiness of some sediments (especially very muddy ones), when the sediment is withdrawn residues remain on the inner walls of the sampling tube, creating the possibility that deeper samples might become contaminated with residues from shallower ones. To eliminate the risk of contamination caused by contact with the sampling tube, it is recommended, where possible, that a ring made of an inert material (metal, plastic or glass, depending on the analysis to be carried out) slightly smaller in diameter than the sampling tube be used to cut away the outermost layer (which should be at least 0.5 cm thick) along the circumference of each core section.

It is recommended to use nitrile gloves (preferably talc-free) when handling samples and to use only plastic when handling heavy metal subsamples. Only instruments made of glass and stainless steel and aluminium foil should be used when handling samples to be analysed for organic pollutants.

Once the cutting of each subsample is begun, the characteristics of the sediment should be recorded immediately using the core characterization form (section 5.3). It is recommended that photographs be taken of each section, if possible.

It is recommended to pick out any macroremains, such as bits of wood, rocks and shells, found along the core, using tweezers made of (a) plastic, for core I, or (b) stainless steel, for sediment to be analysed for organic compounds in core II. The macroremains are to be stored in nylon bags and the depth at which they were found is to be recorded on the core characterization form (Annex I in the supplementary files). Whenever shells of organisms are encountered, it is recommended that the species be identified.

#### 4.6. PROCESSING PROCEDURES FOR EACH TYPE OF CORE

##### 4.6.1. Core I: radionuclides, metals and geochemical parameters

Use a plastic spatula to transfer fresh sediment samples into plastic bags of a known weight (i.e. that have been tared). Weigh the wet sample on scales having a precision of 0.001 g or better. Dry the samples and determine the percentages of moisture and loss on ignition (section 4.2. and Annexes III.1 and III.2 in the supplementary files).

One to two grams of UNGROUND sediment are set aside for grain size analysis by laser diffraction. The rest of the sample is ground in a mortar, preferably one made of agate, to be distributed to the analytical laboratories that are to perform the analyses. Before the mortar is used it must be washed using phosphate-free soap and rinsed under running water, then rinsed once with distilled water and once with diluted 2M HCl, and then finally rinsed three times with deionized water to remove all acid residues. To reduce the risk of contamination between samples, the deeper layers should be ground before the more recent ones. It is also recommended to clean the agate mortar thoroughly between samples and to wash it each time the core is changed.

Aliquots for later analyses are placed in plastic bags of a size suitable for the quantity of sample required for each analysis. (See Table 2 in section 8 for the minimum and optimum amounts of sediment required for the analyses.)

It should be noted that the minimum amount is for sediment core sections that do not contain the optimum amounts of sediment for each analysis owing to high moisture content. It should be emphasized, however, that it is nonetheless important to separate out the optimum amount wherever possible.

##### 4.6.2. Core II: organic pollutants

If the sediment comes into contact with plastic, it could be contaminated by phthalates, which are compounds used to increase the flexibility of many plastics, such as PVC. The sediment used to analyse the organic pollutants is therefore only to be handled with glass or metal instruments. It is recommended to avoid contact between the sediment and the walls of the sampling tube. The sample should therefore be taken from the middle of the core, separating it from the outside of the core using an O-ring made of Teflon or glass (a glass container with a diameter smaller than that of the core can also be used).

The wet sample is placed in pre-weighed (tared) aluminium foil containers (or sheets) and the wet weight recorded. The sample is dried until it reaches a constant weight and then the moisture content is calculated (see section 3.2.). The dry samples are broken up in a porcelain mortar for distribution.

The sediment aliquots that are to be analysed for organic compounds are placed in aluminium foil containers or sheets (precalcinated at 450°C for 4 h). They are then wrapped and put in

plastic bags for distribution. Table 2 (section 8) shows the minimum and optimum amounts of sediment required for the analyses. Sediment samples to be analysed for organic compounds should be kept refrigerated (at 4°C) until they are analysed.

#### **4.6.3. Core III: the control sample**

Core III (for control) is stored, after being cut and dried, for use in case additional analyses are needed. This core is processed for use in analyses both for metals and organic compounds. The samples from each section are therefore divided into two aliquots: (a) one to be used for analysis of organic compounds, which is taken from the middle of the core using a glass container; and (b) the other, to be used for analysis of metals, for which the remaining sediment is collected using plastic utensils. Both must be properly coded (see section 6).

The samples to be analysed for metals are placed in plastic bags, and the samples to be analysed for organic compounds are placed in aluminium foil containers (or sheets) (see sections 5.6.1 and 5.6.2). In both cases, the wet weights are recorded and the samples are dried, in an oven at a temperature below 45°C until a constant weight is reached, in order to determine the moisture content (see section 3.2.).

Once dried, the samples are broken up in a porcelain mortar and kept as a control for analyses performed later.

#### **4.7. STORING THE SAMPLES**

It is important that dried samples to be analysed for heavy metals be stored in a dehydrator to prevent them from absorbing moisture from the surroundings while awaiting analysis. The laboratories must make sure that the samples are dry and if they are not, the material must be re-dried before being analysed.

Dry sediment samples to be analysed for organic compounds should be kept refrigerated at 4°C until they are analysed. In analytical laboratories the samples should also be kept refrigerated until they undergo extraction; sample extracts should be kept at 4°C until they undergo instrumental analysis, after which they should be stored in a dark place at -20°C. It is recommended that any extracts left over from the analysis of samples and any remaining sediment be kept frozen until the end of the study.

### **5. KEEPING RECORDS OF SAMPLES**

In order to have basic information about the location and conditions at the sampling site at the time of collection, the appropriate collection form must be filled out manually.



### 5.1. SURFACE SEDIMENT COLLECTION FORM

Basic information for surface sediment sampling (Annex I in supplementary files) includes the following data:

- Date (day, month, year, time of day);
- Names of those responsible for the campaign and for taking the samples;
- Station number;
- Geographical location (coordinates);
- Depth of the sampling site.

The remarks section is used to note down any details concerning specific identifying characteristics of the sample (river mouth, colour, odour, presence of shell remains in the sediment, etc.).

Where possible, it is useful to have additional data such as temperature, salinity and dissolved oxygen, which can be measured using a multiparameter probe, as well as weather information (air temperature, wind speed and direction) if the vessel has a weather station.

### 5.2. SEDIMENT CORE COLLECTION FORM

Basic information for the sampling of sediment cores is recorded using the corresponding form found in the supplementary files in Annex I, and includes the following data:

- Geographical location (coordinates);
- Date and time of sampling (day, month, year, time of day);
- Hydrographic data (direction and speed of currents and tides, temperature, etc.);
- Weather information (wind direction and speed);
- Sample identification (sampler or cylinder used, core code, core length);
- Names of those responsible for the campaign and for taking the samples.

The remarks section is used to note the depths at which there are changes in colour and other external macroscopic features observed (shells, voids, bubbles, etc.).

### 5.3. SEDIMENT CORE CHARACTERIZATION FORM

Sediment characteristics, such as odour, apparent texture of the sample, colour (according to the Munsell chart) and the presence of macroremains, are to be recorded using the corresponding form in Annex I (in the supplementary files). The wet, dry and calcined weights are also entered on this form so that the moisture content values and percentage of loss on ignition can be calculated for each sample.

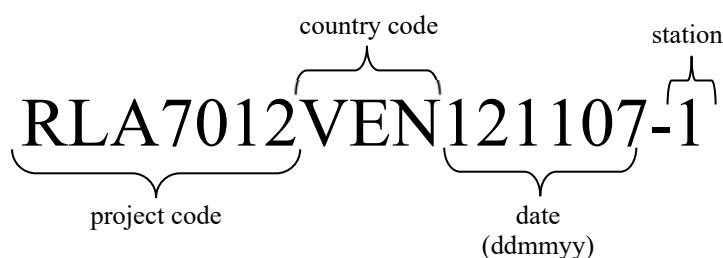
## 6. CODING SAMPLES

In order for each sample to have a unique identifier, a general code is assigned composed of the project code, country code, date collected (ddmmyy) and sampling station. This is then extended to distinguish between surface sediment samples (section 6.1.), sediment cores (sections 6.2. and 6.3.) and samples to be distributed for laboratory analysis (section 6.4.).

The country codes for the project were: COL (Colombia), COS (Costa Rica), CUB (Cuba), DOM (Dominican Republic), GUA (Guatemala), HAI (Haiti), HON (Honduras), JAM (Jamaica), MEX (Mexico), NIC (Nicaragua), PAN (Panama) and VEN (Bolivarian Republic of Venezuela).

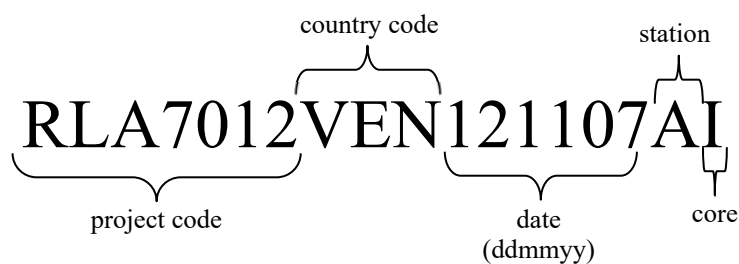
### 6.1. SURFACE SEDIMENTS

Surface sediment samples were labelled as follows: project code, country code, date (ddmmyy) and station (Arabic numeral), as in the example below:



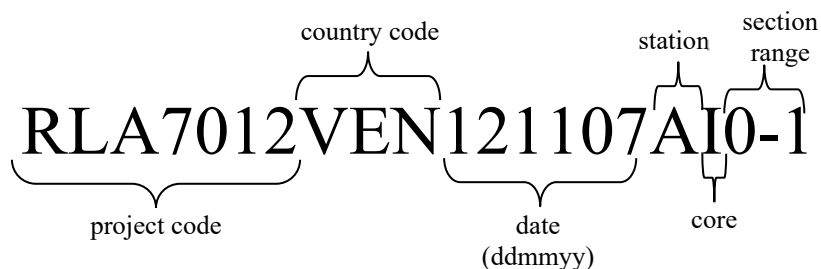
### 6.2. CODES FOR SEDIMENT CORES IN THE FIELD

The cores were labelled as follows: project code, country code, date (ddmmyy), station (letter) and core number (Roman numeral), as in the example below:



### 6.3. CODES FOR THE SECTIONS OF A SEDIMENT CORE

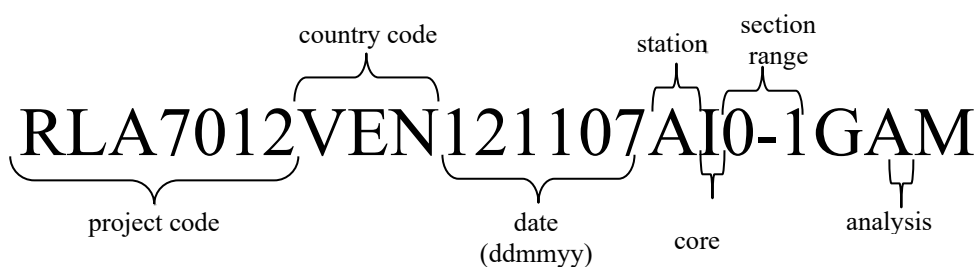
Using the same format, sediment core sections were coded by appending the section range (in cm) after the core code, as in the example below:



This code is to be recorded in the core characterization form (see Annex I, in supplementary files).

#### 6.4. CODES OF SEDIMENT ALIQUOTS FOR ANALYSIS

Once the dry samples were weighed, they were divided according to the analyses to be carried out. The sediment aliquots from each core section were coded as follows: a code was appended after the section range code identifying the type of analysis to be conducted (see Table 2, section 8). An example is shown below, using a surface sample (section range 0–1 cm) for gamma spectrometry analysis (GAM) from project RLA/7/012:



In order to prevent any labels from becoming detached or damaged owing to moisture or contact with the sediment, it is recommended that the labels be placed between the two bags in which the subsample is stored.

### 7. <sup>210</sup>Pb CHRONOLOGY

It may be difficult to collect an undisturbed sediment core in the coastal zone owing to all the possible causes of sediment disturbance (e.g. high levels of biological activity, physical mixing, etc.). Before starting any analysis of pollutants, it is therefore recommended that one make sure that a given sediment core is usable for purposes of <sup>210</sup>Pb geochronology.

#### 7.1. CRITERIA FOR EVALUATING CORES FOR <sup>210</sup>Pb DATING

LOI<sub>550</sub> data are a good approximation of sediment organic matter concentrations [6], and the depth profiles of LOI<sub>550</sub> can be used to assess roughly whether the organic matter concentrations follow the typical curve for exponential decay due to bacterial decomposition [8].

Typical profiles of organic matter degradation can normally only be observed in a sedimentary column that has had minimal disturbance. A profile of LOI<sub>550</sub> concentrations matching this curve would therefore indicate that there has been no mixing in the sediment core and there is a good chance of dating it reliably using the <sup>210</sup>Pb method. It is recommended that the selection of a core for <sup>210</sup>Pb dating be based on the LOI<sub>550</sub> data from core I from each sampling station.

## 7.2. SELECTING THE CORE FOR <sup>210</sup>Pb ANALYSIS

Based on the assumption described in section 7.1., the <sup>210</sup>Pb analysis for dating purposes is performed on core I from the sampling station with an LOI<sub>550</sub> profile that best approximates an exponential relationship between decay and depth.

## 7.3. <sup>210</sup>Pb ANALYSIS STRATEGY

It is recommended to perform an initial test run of analyses at 5 cm depth intervals along the sediment core (i.e. in a core that is 50 cm deep, 10 samples are analysed). Using these data, a preliminary assessment can be made of the <sup>210</sup>Pb profile as a function of depth and the feasibility of obtaining reliable dating results using this core. The <sup>210</sup>Pb profile obtained should be comparable to exponential decay, and if so, all the sections where excess <sup>210</sup>Pb is present are to be analysed.

Geochemical and pollutant analyses should only be carried out once the <sup>210</sup>Pb profile has been confirmed as suitable for obtaining reliable dating results.

## 7.4. DISTURBED CORES

Fig. 2 shows a flow chart describing the proposed workflow for sediment cores. If it is determined that core I from site A is not datable because it has been disturbed, a <sup>210</sup>Pb analysis is to be performed of core I from site B following the same procedure, as described in sections 8.2. and 8.3. If core I from site B is also found to have been disturbed, core I from site C is to be analysed.

# 8. TESTS TO BE CARRIED OUT

Before starting to analyse sediment samples, it is recommended that tests be carried out using reference materials to verify the method's precision and accuracy. A form and a sample of the report of the analysis results are included in Annex II (supplementary files) and should be presented in the International System of Units.

## 8.1. RECOMMENDED ANALYSES

The methods listed below, which were used in project RLA/7/012, are described in detail in Annex III (supplementary files); the minimum and optimum quantities of material required for each analysis are given in Table 2 at the end of this section.

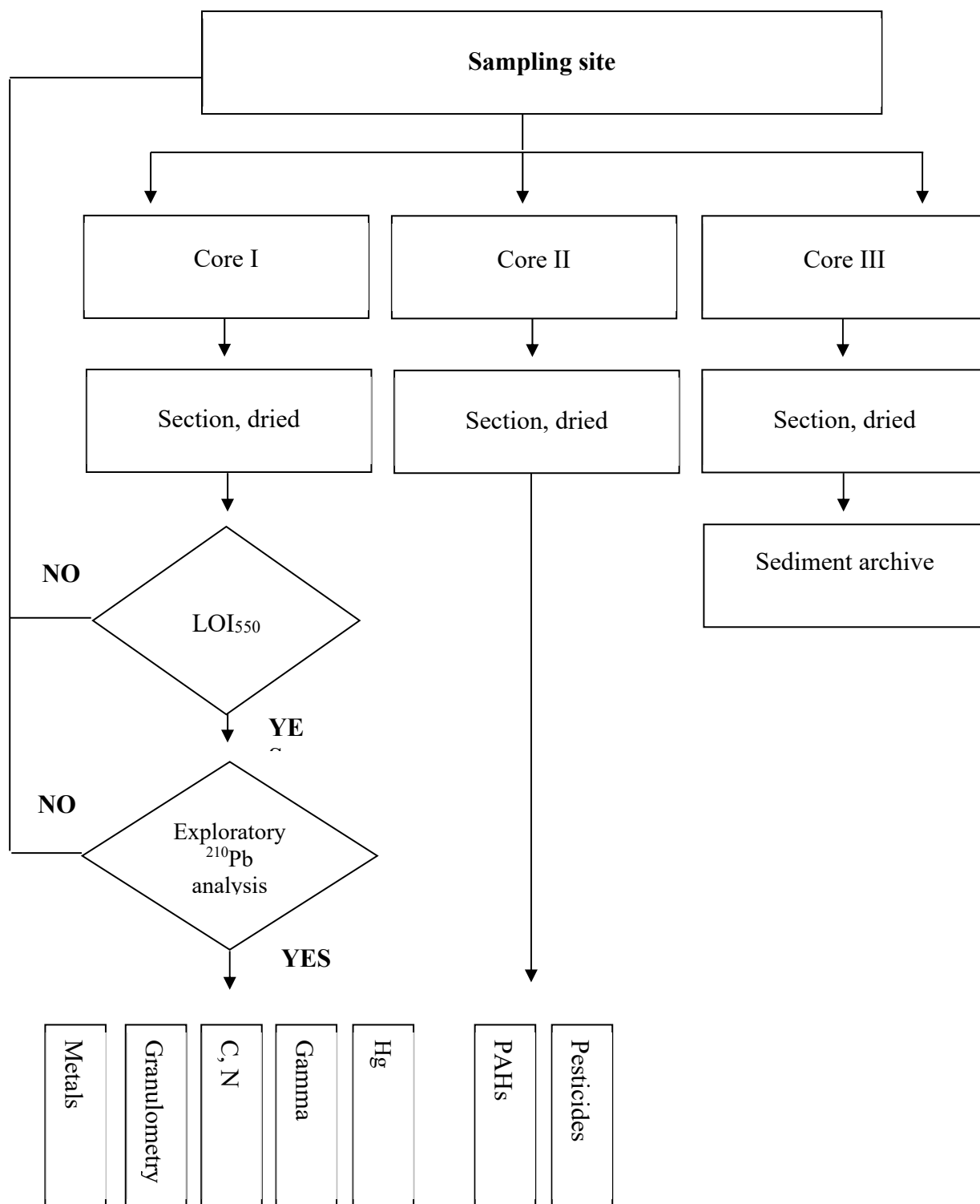


FIG. 2. Flow chart of sample processing.

### **8.1.1. Moisture content**

This is used to calculate the in situ density and porosity of the sediment, which are variables related to sediment compaction — a factor that can affect the accuracy of  $^{210}\text{Pb}$  dating, given that the thickness of the core sections is reduced as a result of having been buried. The total dry weights of the core sections, which are obtained when estimating the moisture and the volume of each one, are used for an accurate calculation of the mass depth ( $\text{g}/\text{cm}^2$ ). This is a fundamental parameter in  $^{210}\text{Pb}$  dating that is essential in adjusting for the effects of compaction on  $^{210}\text{Pb}$  profiles and pollutants[5].

### **8.1.2. Loss on ignition**

This provides an estimate of the sediments' organic matter content. Although it is usually known to overestimate organic matter content owing to the loss of structural water in clay minerals, it is a simple and inexpensive technique that quickly reveals the relationship between organic matter concentration and depth in the cores, which is used as a preliminary indicator to identify cores to be selected for  $^{210}\text{Pb}$  dating.

The importance of the LOI<sub>550</sub> analysis in this project's working strategy is based on three empirical observations: (a) the availability of suspended organic matter in the water column is one of the main factors regulating  $^{210}\text{Pb}$  flow to sediments; (b) buried organic matter in sediments decomposes as a result of bacterial action; (c) organic matter concentrations decrease over time owing to mineralization and  $^{210}\text{Pb}$  activities decrease over time owing to radioactive decay, and both quantities increase exponentially with depth in undisturbed sediment [7]. If the LOI<sub>550</sub> depth profiles show an exponential relationship to depth, sediment disturbance is probably minimal, and therefore these cores are more likely to provide usable  $^{210}\text{Pb}$  profiles for dating purposes.

### **8.1.3. Grain size distribution**

This shows the percentage of the three main grain size fractions (sand, silt and clay) and is useful both in explaining  $^{210}\text{Pb}$  outliers in anomalous profiles (other than exponential decay) and in normalizing heavy metal values. Annex III (in the supplementary files) contains recommendations on preparing samples for grain size analysis.

### **8.1.4. Element composition**

This analysis reveals the concentration of elements that are indicators of diagenetic processes in the sedimentary column (Fe, Mn), deposition of terrigenous material in sediments (Al, Ti) and metal pollutants such as As, Cd, Hg, Cu, V, Pb and Zn.

### **8.1.5. Total carbon, total nitrogen, total inorganic carbon and total organic carbon**

An analysis of C and N concentrations and flows is useful in identifying the process of the nutrient enrichment of water bodies (eutrophication trends) and to assess the origin of organic matter in sediments (by calculating the C/N ratios).

### **8.1.6. $^{210}\text{Pb}$**

$^{210}\text{Pb}$  is one of the radioactive tracers used to obtain the geochronology of sediment cores. In project RLA/7/012,  $^{210}\text{Pb}$  activity was estimated using alpha spectrometry by determining the  $^{210}\text{Po}$ , assuming radioactive equilibrium between them. The analytical method [9], adapted from [10], is described in Annex III (in the supplementary files).

### **8.1.7. $^{226}\text{Ra}$ and $^{137}\text{Cs}$ by gamma ray spectrometry**

The  $^{210}\text{Pb}$  dating method requires determining  $^{226}\text{Ra}$  and corroborating the dates using the artificial radionuclide  $^{137}\text{Cs}$ . High-resolution, low-background gamma spectrometry can be used for the determination of both radionuclides.

### **8.1.8. Total mercury**

Total mercury can be quantified using direct mercury analysers (AMA) following the US EPA reference method [11].

### **8.1.9. Polycyclic aromatic hydrocarbons**

PAHs can be analysed using gas chromatography (GC-FID or GC-MS) depending on each laboratory's capabilities.

### **8.1.10. Pesticides (organochlorine hydrocarbons)**

Pesticide analyses can be performed using gas chromatography (GC-ECD).

## **8.2. OPTIONAL ANALYSES**

### **8.2.1. Temperature and salinity**

These measurements describe the environment in the area of interest and are obtained using a multiparameter probe.

### **8.2.2. Dissolved oxygen**

This reveals the level of oxygenation at the bottom of the sea, enabling a better understanding of the profiles of redox-sensitive metals such as Fe, Mn and/or Cd; its is measured using a multiparameter probe.

### 8.2.3. Mineral composition

Information on the main mineral components in sediments, e.g. the distribution of clay minerals, is used to identify the origin of the sediments and is obtained using X-ray diffraction analysis.

### 8.3. ANALYTICAL QUALITY CONTROL

Laboratories should routinely test duplicate samples, reagent blanks and certified reference materials, and it is important that they participate in intercalibration and intercomparison exercises.

It is also recommended that laboratories apply the same analytical procedure to all samples tested (including blanks, duplicates, etc.) in order to maximize the reproducibility of results.

TABLE 2. METHODS OF ANALYSIS AND ALIQUOTS REQUIRED

Analysis	Unit	Method	Code	Minimum (g)	Optimum (g)
<i>Core I: Radionuclides, metals and geochemical parameters</i>					
Moisture	%	Oven (45°C)	MOI	–	Total
Loss on ignition	%	Muffle (550°C)	LOI	0.5	0.5
C, N	%	Elemental analyser	C–N	1.0	1.5
Metals	%	X-ray fluorescence	XRF	4.0	5.0
<sup>210</sup> Pb, <sup>137</sup> Cs	Bq/kg	Gamma spectrometry	GAM	20	25
<sup>210</sup> Pb ( <sup>210</sup> Po)	Bq/kg	Alpha spectrometry	ALP	0.5	1.0
Grain size	%	Laser beam diffraction	GRA	1.0	2.0
<i>Core II: Organic compounds</i>					
PAHs	mg/kg	GC–FID, GC–MS	PAH	5	10
Pesticides	mg/kg	GC–ECD	PES	5	10
Mercury	mg/kg	DMA	DMA	0.5	2.0
<i>Optional analyses</i>					
Water temperature	°C	Probe	T	–	–
Water salinity	–	Probe	S	–	–
Dissolved oxygen	mg/L	Probe	DO	–	–



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The supplementary files for this guide can be found on the web page for this guide at:  
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