



Analytical methods to determine ^{210}Po and ^{210}Pb in marine samples

Maria Assunta Meli, Donatella Desideri*, Carla Roselli, Laura Feduzi

Department of Biomolecular Sciences, Urbino University "Carlo Bo," P.zza Rinascimento 6, 61029, Urbino, Italy

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ABSTRACT

This paper describes the methods of sampling, preconcentration, chemical separation and final count of ^{210}Po and ^{210}Pb present in different marine matrices to determine their background levels in a marine ecosystem. Complex, time consuming and selective radioanalytical methods have been used to prepare final clear sources for alpha spectrometry and beta count; in fact, gamma spectrometry, a method direct and non destructive, cannot be taken into account because the ^{210}Pb activity is very low which does not allow to carry out sufficiently accurate measurements and ^{210}Po is not a gamma emitter but it emits only alpha particle at 5.40 MeV. The results of ^{210}Po and ^{210}Pb activity concentrations obtained in different marine samples collected in the first two campaigns are still very few to discuss about the ^{210}Po and ^{210}Pb behavior in marine environment.

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1. Introduction

Naturally occurring ^{210}Po and its grandparent ^{210}Pb are members of ^{238}U decay series. ^{210}Po is an alpha radioactive emitter which decays ($T_{1/2}$: 138.4 days) to stable lead. In the marine environment, ^{210}Po is largely produced from the decay of ^{210}Pb ($T_{1/2}$: 22.3 years) deposited from the atmosphere. A small amount of ^{210}Po in the seawater originates from the atmospheric deposition of polonium itself [1,2].

Many authors determined ^{210}Po in environmental surveys but these measures are not accompanied by that of ^{210}Pb , perhaps because of the complexity of the methods, but the importance of measuring ^{210}Pb in addition to ^{210}Po must not be ignored [3,4]. Since ^{210}Pb is a continuing source of ^{210}Po , knowledge of its content is necessary for an accurate determination of the ^{210}Po content at the sampling time. On the other hand, it is necessary to calculate the ^{210}Po content at equilibrium, that is, when polonium input (whether technological or natural) has ceased and unsupported ^{210}Po has decayed. Furthermore, the activity ratio $^{210}\text{Po}/^{210}\text{Pb}$ provides information about the time scale of ^{210}Po incorporation. Therefore, it is very important to dispose of reliable radioanalytical methods for the accurate determination of these natural radionuclides in the marine environment. For the ^{210}Po and ^{210}Pb determination non-destructive and destructive methods could be used.

The principal non destructive method is the gamma spectrometry that involves a direct determination of radionuclides in the original sample. It is possible to determine simultaneously many radionuclides by this technique without any specific pre-treatment of the sample. Nevertheless, it is impossible to determine directly all the radionu-

clides of interest because some of them, as ^{210}Po , are not gamma emitters. It is not possible to use the gamma emission of other members of ^{238}U series because a secular equilibrium for all members of the series cannot be assumed [5].

As far as the ^{210}Pb determination is concerned, a direct γ -spectrometry of the sample can be used taking into account the 46.5 keV photons representing only a small fraction of the total disintegrations [6]. Many environmental samples, as those of marine ecosystem, show such a low concentration which do not allow to carry out sufficiently accurate measurements, but in consideration of the advantages of direct methods, many efforts have been made to optimize this technique by using detectors particularly sensitive to low energies or by preparing very compact sources. When the above equipment and methodologies are not available, it is possible to determine indirectly ^{210}Pb by measuring the α or β emissions of its daughters ^{210}Po or ^{210}Bi , respectively [7–10].

Therefore, to determine ^{210}Pb and ^{210}Po it is often necessary to use the radiochemical methods. A radiochemical method is a destructive technique; it consists in measurements of the radionuclides after their separation (by extraction chromatography, precipitation, electrodeposition, etc.) from the solution coming from the complete dissolution of the sample. Radiochemical separations play an important role when alpha and beta emitting radionuclides must be determined in complex environmental matrices. This technique is certainly more sensitive in comparison to the gamma spectrometry and it can detect radioactivity at very low levels. However, an extensive sample preparation, together with separation and purification procedures is often required to achieve accurate results. When the matrix is very complex (sediments, particular matter, etc.) many separation steps must be followed to achieve the complete purification of a radionuclide to be determined from interfering elements whose weight-sare often 10^{10} – 10^{12} times greater than that of the radionuclide. In

* Corresponding author. Tel.: +39 0722303307; fax: +39 0722303306.
E-mail address: donatella.desideri@uniurb.it (D. Desideri).

these quite complex radiochemical procedures it is necessary to add a stable or radioactive tracer to calculate the final chemical yield, sample by sample. In addition, it is often required to set-up a specific method for each particular radioisotope in each type of matrix [11].

Because ^{210}Po and ^{210}Pb concentration in sea samples is very low, it is also needed a) to collect large quantities of sample and to utilize peculiar and selective preconcentration techniques before the radiochemical separation and b) to measure the final source by count systems, as the alpha spectrometry for ^{210}Po and the beta counter for ^{210}Pb , having a low background and an acceptable efficiency.

This paper describes the methods of sampling, preconcentration, chemical separation and final count of ^{210}Po and ^{210}Pb present in different marine matrices.

2. Experimental

2.1. Samples and sampling

The samples taken into account and representing the different components of a marine ecosystem are the following: sea water, surface sediment, total particulate ($>0.45\ \mu\text{m}$) matter, phyto and zooplankton (organic matter).

The samples are collected in the central Adriatic Sea onboard the “Athena” of Urbino University equipped by radar, GPS, and a probe to monitor the physics and chemical parameters in surface water and along the water column.

To collect water samples (10 l for ^{210}Po and 100 l for ^{210}Pb) at different depths and at different distances from the coast, a bottle of Niskin is used (Fig. 1) and clean polyethylene samplers were used to store these.

For the simultaneous sampling of phyto and zooplankton at 1500 m from the coast, a particular system, shown in Fig. 2, is realized in our laboratory; this is constituted by 2 nylon nets positioned one inside the other, equipped with a collecting beaker (internal net for zooplankton collection: $200\ \mu\text{m}$ of mesh size and 100 cm of length; external net for phytoplankton: $50\ \mu\text{m}$ of mesh size and 150 cm of length), and fixed to a single ring (diameter, 40 cm). The system is equipped also with a fluximeter to know the sea water volume.

The particulate matter (organic and inorganic) is obtained by filtering, in the laboratory, the water samples (100 l) through 0.45 membrane filter paper (i.d.: 9 cm). A bucket Van Veen is used for surface sediment collection at 3000 m from the coast (Fig. 3).

2.2. Preconcentration methods

2.2.1. Water samples

After filtration to separate particulate (>0.45) from dissolved phase (<0.45), a known activity of ^{209}Po as the yield internal standard

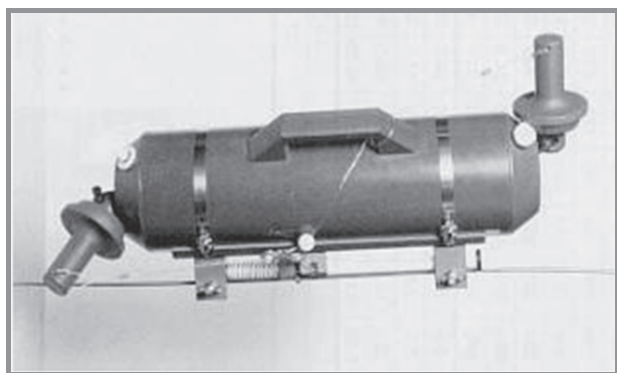


Fig. 1. Bottle of Niskin for sampling of sea water.

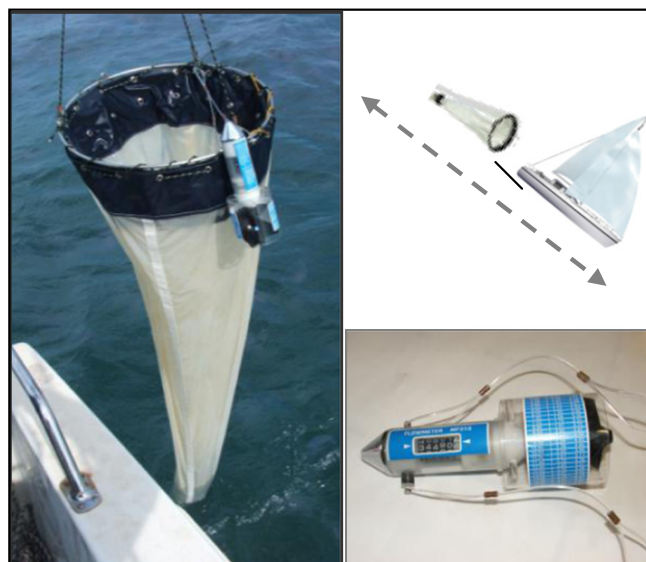


Fig. 2. Phyto and zooplankton sampling system and fluximeter.

is added to samples (10 l) for polonium determination. The samples are acidified by addition of concentrated HCl until pH 1; to samples for lead determination (100 l), a known quantity of Bi^{3+} as yield internal standard and Pb^{+2} as carrier are added. 125–750 mg of Fe^{3+} is added to all samples (both 10 l and 100 l) to coprecipitate, in alkaline medium, the radionuclides of interest as hydroxides. After digestion for 24 h, the supernatant was siphoned off and the remaining precipitates are centrifugated and dissolved with concentrated HCl. The solutions were mineralized with H_2O_2 at 85–90 °C. The solutions for polonium are diluted until 1 M and boiled with a few milligrams of ascorbic acid to reduce Fe^{3+} .

In the solution for lead, after addition of 50 ml of concentrated HCl, Fe^{3+} is separated by a double extraction with methyl-isobutyl-ketone.

2.2.2. Particulate matter

All the filters coming from the filtration of 100 l of sea water are dissolved and mineralized with concentrated HNO_3 and H_2O_2 . The nitric solution is evaporated until dryness and the residue is boiled with concentrated HNO_3 and H_2O_2 at 85–90 °C, evaporated, re-dissolved and evaporated again. The residue is treated with concentrated HF and then 3 times with concentrated HCl, dissolved in 1 M HCl until 250 ml and filtered. The solution was subdivided in 2 fractions: 25 ml for polonium and 225 ml for lead determination. A

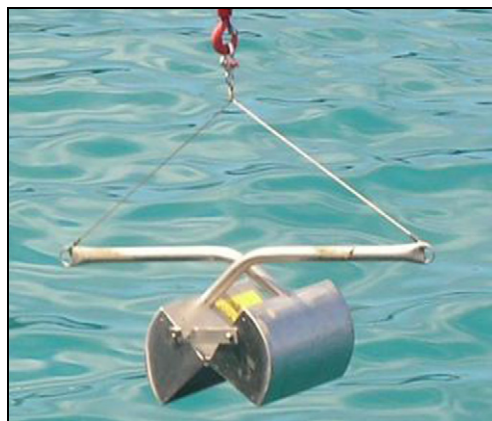


Fig. 3. Bucket Van Veen.

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