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ACTIVATION ANALYSIS AS A METHOD FOR
TRACING SUSPENDED SEDIMENTS

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LEGAL NOTICE

ACTIVATION ANALYSIS AS A METHOD

FOR TRACING SUSPENDED SEDIMENTS

by

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and

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ABSTRACT

Neutron Activation Analysis was applied to the problem of sedimentation in the Mayaguez Bay. The method was chosen as an attempt to eliminate the cumbersome chemical methods normally employed, and because it offered the opportunity to increase the sensitivity greatly.

The masking effect of Na^{24} and Cl^{33} were eliminated by filtering the samples through 0.45 μ millipore filter paper. The papers were allowed to dry in a closed hood to minimize airborne contamination and dust from entering the samples. The weight of the particulate matter in the filter paper was determined by weighing the papers before and after filtration.

The filter papers were placed in one inch polyethylene vials and irradiated for 3 sec, together with aluminum standards. They were analyzed 30 sec later, using a NaI (Tl) crystal. The samples and standards were counted in alternate order for 40 sec live time, for a period which did not exceed 5 min. Aluminum was found to be present in all the samples in different concentrations.

The results demonstrated that aluminum may be used for tracing the sediments contributed by river waters, offering the opportunity of determining their distribution pattern and settling rate.

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INTRODUCTION

Many new techniques have developed almost as by-products of Nuclear Power, Activation Analysis is one of them. "Activation" by some type of nuclear reaction is used to produce a radioisotope from the element to be determined. There are two major advantages in using activation analysis: first, since the instrumentation available nowadays allows the detection and measurement of very small amounts of radioactivity, this proves to be a very sensitive method, second, since the radioisotope formed decays with its own characteristic radiations and half-life, it is feasible to make activation analysis very specific. Besides its high sensitivity, activation analysis is a fast and economical method of analysis.

Of all the possible nuclear reactions, the neutron reactions have been most often used. In the analysis of complex mixtures of nuclides the major constituents with high neutron cross-sections mask the spectra of other trace activation products. Most of the methods that are used to analyze complex mixtures by neutron activation employ suitable chemical separations to eliminate the interfering induced activities. If they are done before irradiation, some of the interfering activities would be intensified or new ones would be introduced. In general these chemical separations are time consuming and therefore, if done after irradiation, are not practical when analyzing for short-lived isotopes.

When analyzing for short-lived isotopes entirely instrumental methods must be used. By analyzing the sample immediately after irradiation, very small amounts of these short-lived isotopes can be measured. In this work a technique was developed to measure small amounts of aluminum present in sea water samples.

REVIEW OF LITERATURE

Increasing interest has been shown in purely instrumental methods of neutron activation analysis, and a number of studies not involving chemical separations have been reported.

V.P. Guinn[1] has published a review of some recently developed instrumental methods and techniques which are now in use. Some of these methods employ pneumatic tubes to analyze the samples as short as two seconds after irradiation.

O. V. Anders[3,4] has applied the concept of gamma-ray difference spectrum to suppress the longer-lived components in the gamma spectra of activated samples. The method consists in analyzing the samples shortly after irradiation and also after the short-lived isotopes have decayed. By substracting the second spectrum from the first, the effect of the longer-lived isotopes is almost entirely removed from the gamma-ray spectrum.

D.E. Robertson, L.A. Rancitelli and R.W. Perkins[5] used neutron activation and direct counting techniques to measure concentrations of numerous trace elements in sea water, marine organisms and pelagic sediments.

R.L. Caldwell and W.R. Mills, Jr.[6] have used rapid neutron activation techniques for quantitative determination of Silicon, Aluminum and Magnesium in rocks. The accuracy and reproducibility of the analyses and interferences from other element are given.

R.E. Greene[17] has reviewed the use of activable stable isotopes with various types of activation analysis for tracing. Advantages and disadvantages of the method are given, and several examples of applications

in medicine, pollution studies and hydrology are described.

P. Harremoes[20] has described the use of radioisotopes in tracing the effluent dispersion at a proposed site for sewage disposal. Tracer injections at different sites permit a selection of the most economical site according to the required purification of the sewage.

Neutron Activation Analysis

When a stable nuclide captures neutrons and forms a radioactive product which has relatively low absorption cross section, the rate of change of the number of atoms of the radioactive product will be given by:

$$\frac{dN(t)}{dt} = \frac{N_a W_o \sigma \phi}{A} - \lambda N(t) \qquad (1)$$

where

$$N(t) = \text{Number of radioactive atoms}$$

$$N_a = \text{Avogadro's number}$$

$$W_o = \text{Weight of the stable nuclide}$$

$$\sigma = \text{Neutron activation cross section}$$

$$A = \text{Atomic mass number of stable nuclide}$$

$$\phi = \text{Neutron flux}$$

$$\lambda = \text{Decay constant of the radioactive product}$$

multiplying both sides of equation 4 by $e^{\lambda t}$

$$e^{\lambda t} \frac{dN(t)}{dt} = \frac{N_a W_o \sigma \phi}{A} e^{\lambda t} - \lambda e^{\lambda t} N(t) \qquad (2)$$

$$e^{\lambda t} \frac{dN(t)}{dt} + \lambda e^{\lambda t} N(t) = \frac{N_a W_o \sigma \phi}{A} e^{\lambda t} \qquad (3)$$

Since the left hand side is the derivative of a product;

$$\frac{d}{dt} \left\{ e^{\lambda t} N(t) \right\} = \frac{N_a W_o \sigma \phi \, e^{\lambda t}}{A} \qquad (4)$$

Then integrating, and assuming $N(0) = 0$

$$e^{\lambda t} N(t) = \int_0^t \frac{N_a W_o \sigma \phi}{A} e^{\lambda t'} \, dt' \qquad (5)$$

$$e^{\lambda t} \, N(t) = \frac{N_a W_o \sigma \phi}{A \lambda} (e^{\lambda t} - 1) \tag{6}$$

$$N(t) = \frac{N_a W_o \sigma \phi}{A} (1 - e^{-t}) \tag{7}$$

where $\lambda N(t)$ is the activity of the radioactive product. The activity after irradiation time t and a decay time is given by

$$\text{Act} (t, \tau) = \frac{N_a W_o \sigma \phi}{A} (1 - e^{-\lambda t}) \, e^{-\lambda \tau} \tag{8}$$

and the count rate will be,

$$\text{C.R.} (t, \tau) = \epsilon \, \text{Act} (t, \tau) \tag{9}$$

where ϵ is the efficiency of the detector. For a point source on the axis of a cylindrical scintillator as in Fig. 1 it is given by,

$$\epsilon = \frac{\int_{\Omega o} (1 - e^{-\Sigma x}) \, d\Omega}{\Omega o} \tag{10}$$

where Ωo is the solid angle subtended by the crystal measured from the point source, and Σ is the total absorption coefficient.

Quantitative Analysis

In order to perform quantitative analysis of any sample using activation analysis, we can measure the flux, cross section and efficiency of our system, or compare our samples with known samples or standards. In order to compare the activity of the sample with that of a standard, they must have been exposed to the same neutron energy spectrum and neutron flux, and have similar count rates, thus producing essentially the same detector dead time. To obtain the same detector efficiency, the geometries must also be equal.

ILLUSTRATION FOR CALCULATING EFFICIENCY
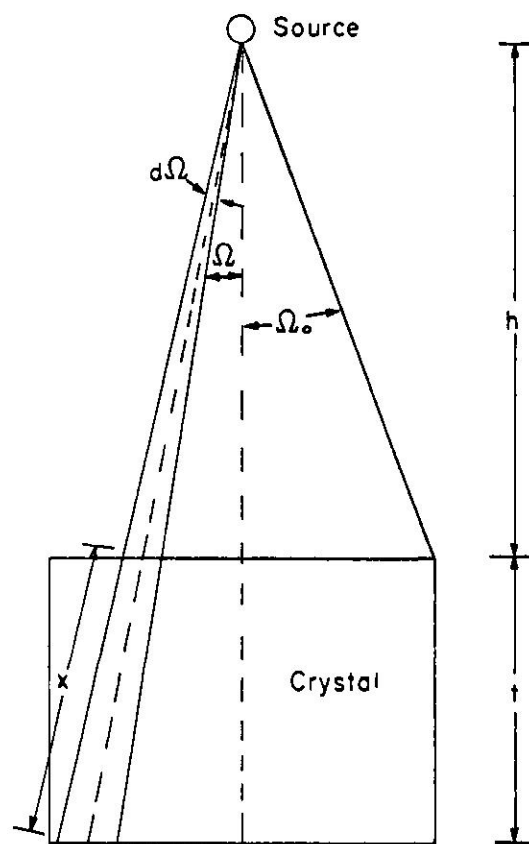
Fig. 1

By irradiating the sample and standard for the same time and essentially in the same place we can assume the same value of the flux and the same neutron energy spectrum. By choosing the weight of the standards so that their count rates will be similar to that of the samples, similar dead times can be obtained. If the standard and the sample are located in the same place relative to the detector, and having essentially the same dead time the same efficiency can be assumed.

Taking the ratio of the count rate of the sample to that of the standard we get,

$$\frac{(CR)s}{(CR)_{STD}} = \frac{\epsilon \, N_a \, W_o \sigma\phi \, (1 - e^{-\lambda t})e^{-\lambda\tau}/ \, A}{\epsilon \, N_a \, W_{STD}\sigma\phi(1 - e^{-\lambda t})e^{-\lambda\tau}/ \, A} \qquad (11)$$

Therefore, if the decay time is the same

$$\frac{(CR)s}{(CR)_{STD}} = \frac{W_o}{W_{STD}} \qquad (12)$$

$$W_o = \frac{(CR)s}{(CR)_{STD}} \, W_{STD} \qquad (13)$$

where W_{STD} is the weight of the standard.

THE EXPERIMENT

Description of Electronic Equipment

1. Scintillation Detectors

A 2" x 2" sodium iodide crystal activated with thallium was used for the detection of gamma rays. It was coupled to a 10 stage,

photomultiplier tube which was operated at 935 volts.

2. Preamplifier

The purpose of the preamplifier is to increase the signal amplitude from the detector and provide impedance transformation; that is, it transforms the voltage which is developed across the small capacity at the input of the preamplifier into an approximately equal voltage across the high capacity output cable. Canberra Industries Model 805 Scintillation Preamplifier was used for these experiments. It is a charge sensitive, all-silicon transistor device which integrates the charge output signals from scintillation/photomultiplier detectors, for presentation to the pulse shaping main amplifier.

3. Amplifier

The function of this instrument is to increase further the signal amplitude from the preamplifier. The gain can be adjusted to obtain the desired energy scale in the analyzer. C.I. Model 810 double delay amplifier was used for this experiment. It accepts the pre-amplified signals from proportional counters, scintillation detectors, or semiconductor detectors. It yields in turn output signals suitable for single channel or multichannel analysis, and for leading edge or zero crossing timing.

4. High Voltage D.C. Supply

This instrument transforms the 115 Volt, 60 C.P.S. A.C. voltage to high D.C. Voltage to be used by the photomultiplier tube. The one used for this experiment was a Fluke Model 409 A, whose ouput ranges to 1,500 volts D.C. with less than .002% ripple and 3ma. output.

5.   Bin and Power Supply

Ortec Model 401 A Modular System Bin was used to provide power for the amplifier. It can provide plus or minus 12, or 24 volts D.C. and 115 A.C. delivered to assigned module connector pins.

6.   Multichannel Analyzer

The Multichannel Analyzer consists of the following elements: (1) the analog to digital converter that associates each input signal with a specific amplitude channel, (2) the memory, or data storage, which contains the information of the number of pulses in each channel, and (3) the oscilloscope, typewriter, and curve plotter which provide for the display of the data which is stored in the memory. The system for this experiment was a Nuclear Data series 2201 system analyzer, which is a completely modular multichannel analyzer.

A block diagram of the electronic equipment is shown in Fig. 2. All the irradiations were done at the P.R.N.C. research reactor at a power level of 1 MW.

Pneumatic Transfer System

1.   Description of the System

The system consists of a pipe through which the "rabbit", (the vehicle where the samples are placed), travels to and from the reactor. The time of irradiation can be adjusted from one second up to thirty minutes. When the rabbit dispatch button is pressed, vacuum is applied at the reactor end of the pipe, thus pulling the rabbit in toward the reactor. When vacuum is applied instead at the other end of the pipe the rabbit is made to return.

2.   Modification Done to the System

Fig. 3 illustrates the modification done to the pneumatic

BLOCK DIAGRAM OF THE ELECTRONIC EQUIPMENT

SAMPLE

γ DETECTOR

PRE-AMPLIFIER

AMPLIFIER

MULTI CHANNEL ANALYZER

HIGH VOLTAGE POWER SUPPLY

BIN POWER SUPPLY

TYPEWRITER

PLOTTER
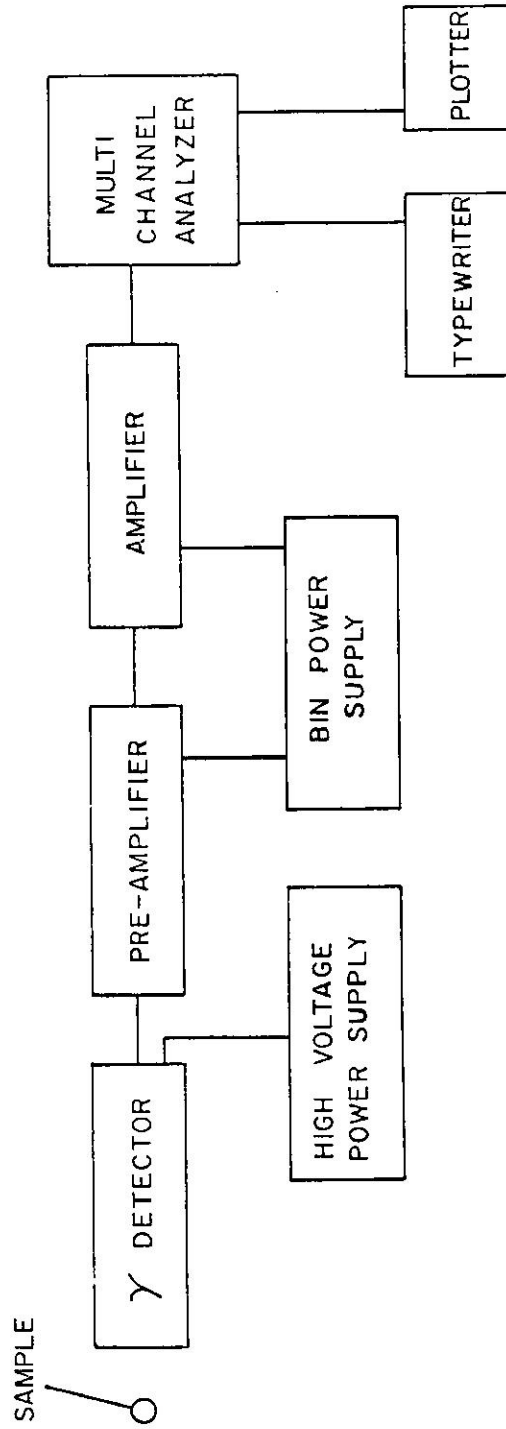
Fig. 2

MODIFICATIONS DONE TO THE PNEUMATIC SYSTEM

To the Reactor

Butterfly
Valve

Switch

Vacuum

Rabbit
Receiving
Box

Short Pipe
Connection
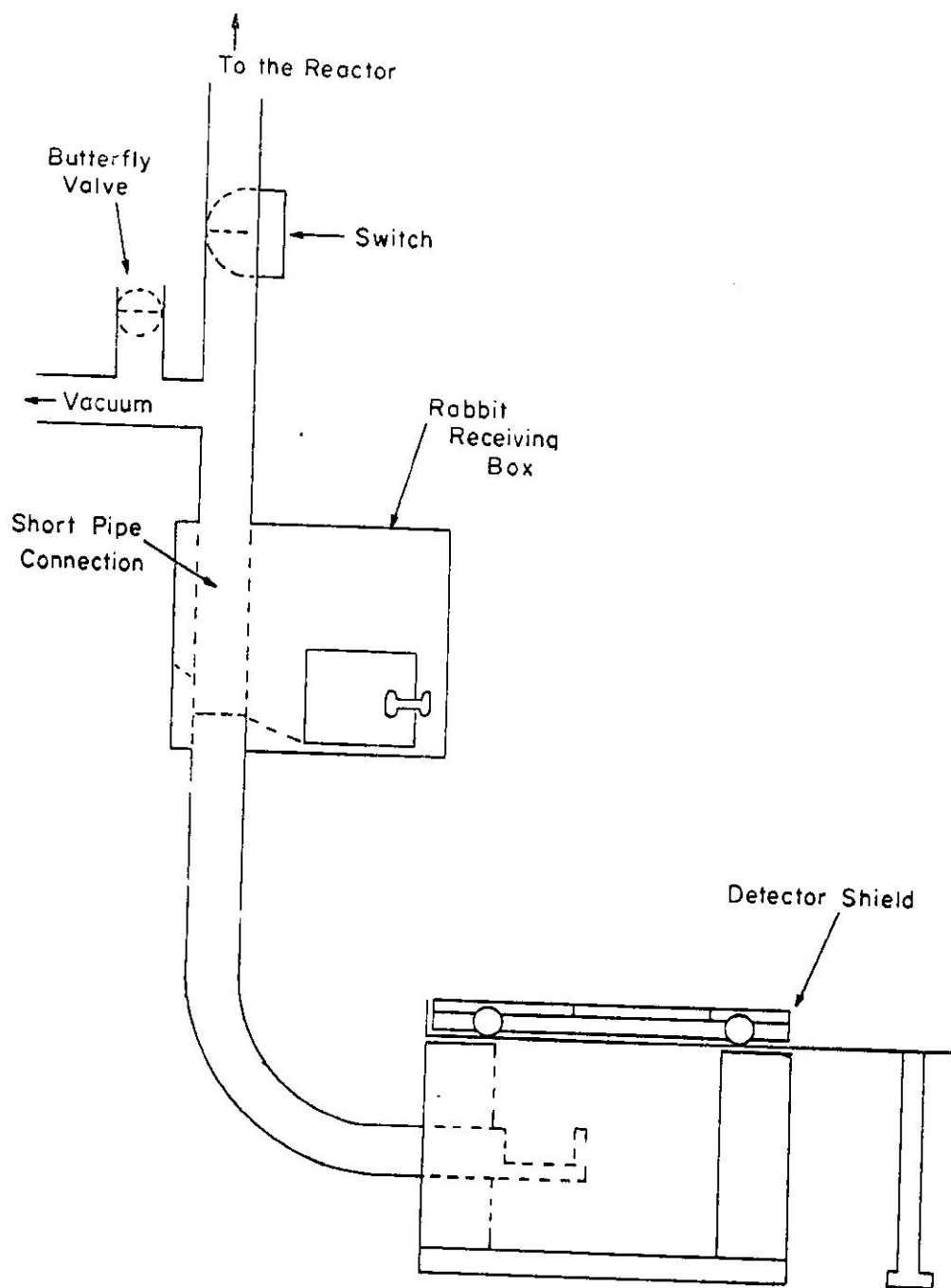
Detector Shield

Fig. 3

transfer system. When the system was set on "rabbit return", the
vacuum applied above the receiving box to make the rabbit return from
the reactor also pulled the rabbit back after it had passed the vacuum
connection, this made the rabbit loose almost all of its speed making
it impossible to reach the end of the pipe. In order to remove the
vacuum effect a butterfly valve was installed in such a manner that it
was activated by the rabbit as it approached the vacuum connection. By
installing a curved pipe below the receiving box the rabbit was trans-
ferred from the reactor directly into the detector shield. A short pipe
which was placed inside the receiving box allowed the rabbit to by-pass
the box.

Detector Shield

The detector shield used for this experiment is shown in Fig. 4.
It was made from solid lead in the form of a hollow cylinder. The
circular walls are four inches thick and have three circular
cavities, two for the detectors and one for the rabbit tube. The two
detector cavities which extend beyond the outer wall of the cylinder
provide space for detectors of different sizes. The walls of the external
cavities are two inches thick and two lead plugs of the same thickness
are located behind each detector.

The inner walls of the shield were covered with .027" of cadmium
and .0625" of copper to absorb the characteristic lead X-rays, which
are produced in the walls of the detector shield.

Since the shield was coupled to the pneumatic system, it had
to be sealed air tight, so that the vacuum would effectively pull the
rabbit from the reactor.
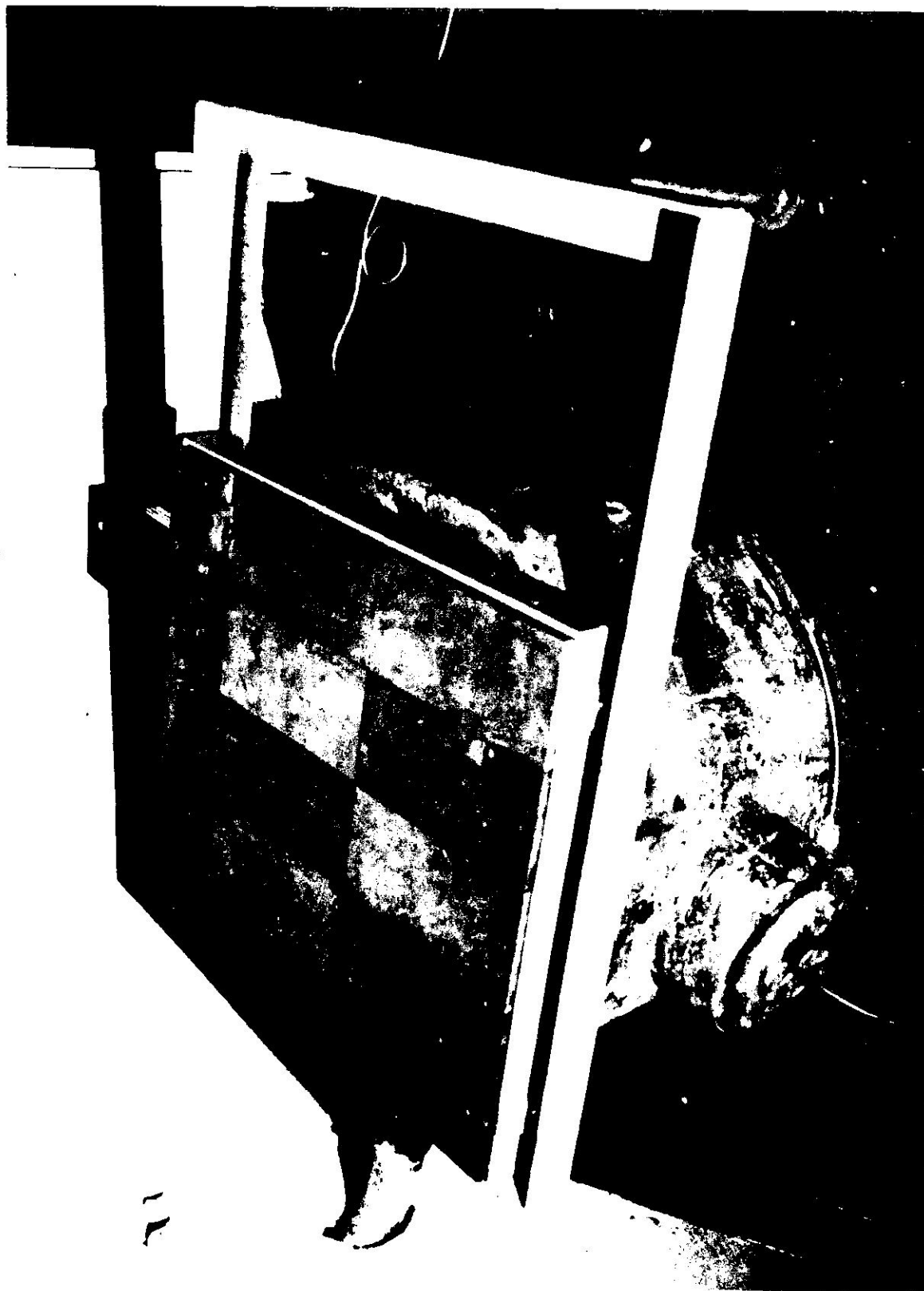
THE DETECTOR SHIELD

Fig. 4

Two styrofoam layers were placed inside the shield to provide support for the detectors. The detector was embedded in the top layer thus remaining in a fixed position.

Figure 5 shows the dimensions of the shield and the relative position of the detector.

D. Sample Collection and Preparation

Sea water samples were collected using the sample collector shown in Fig. 6. Glass bottles were previously washed with 4N Nitric Acid and distilled water. By holding the sample collector at the desired depth a bottle placed in the sample collector will be completely filled with the surrounding water. Samples were collected at different locations and at different depths in the vicinity of a sewage outfall in the Mayaguez Bay.

Since the purpose of this project was to develop a technique, so that the samples could be irradiated and analyzed without chemical separations, 1 ml. samples of the water were irradiated, using small polyethylene vials, and were then directly analyzed. The water samples were irradiated for three seconds and analyzed after thirty seconds of decay. Only Sodium Chloride could be detected as can be seen in Fig. 7 where one such spectrum is shown.

The remainder of the sea water samples were filtered, using 0.45 μ millipore filter paper, measuring one inch in diameter. The filter papers were allowed to dry in a closed hood to minimize airborne contamination and dust from entering the samples. The weight of the particulate matter in the filter paper was determined by weighing the

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DIAGRAM OF THE DETECTOR SHIELD

|←2"→|

DETECTOR

SAMPLE

← 6" →

STYROFOAM
LAYERS

11"

2 1/2"

|←2"→|

|←———— 14" ————→|

|←———————— 22" ————————→|

|←2"→|
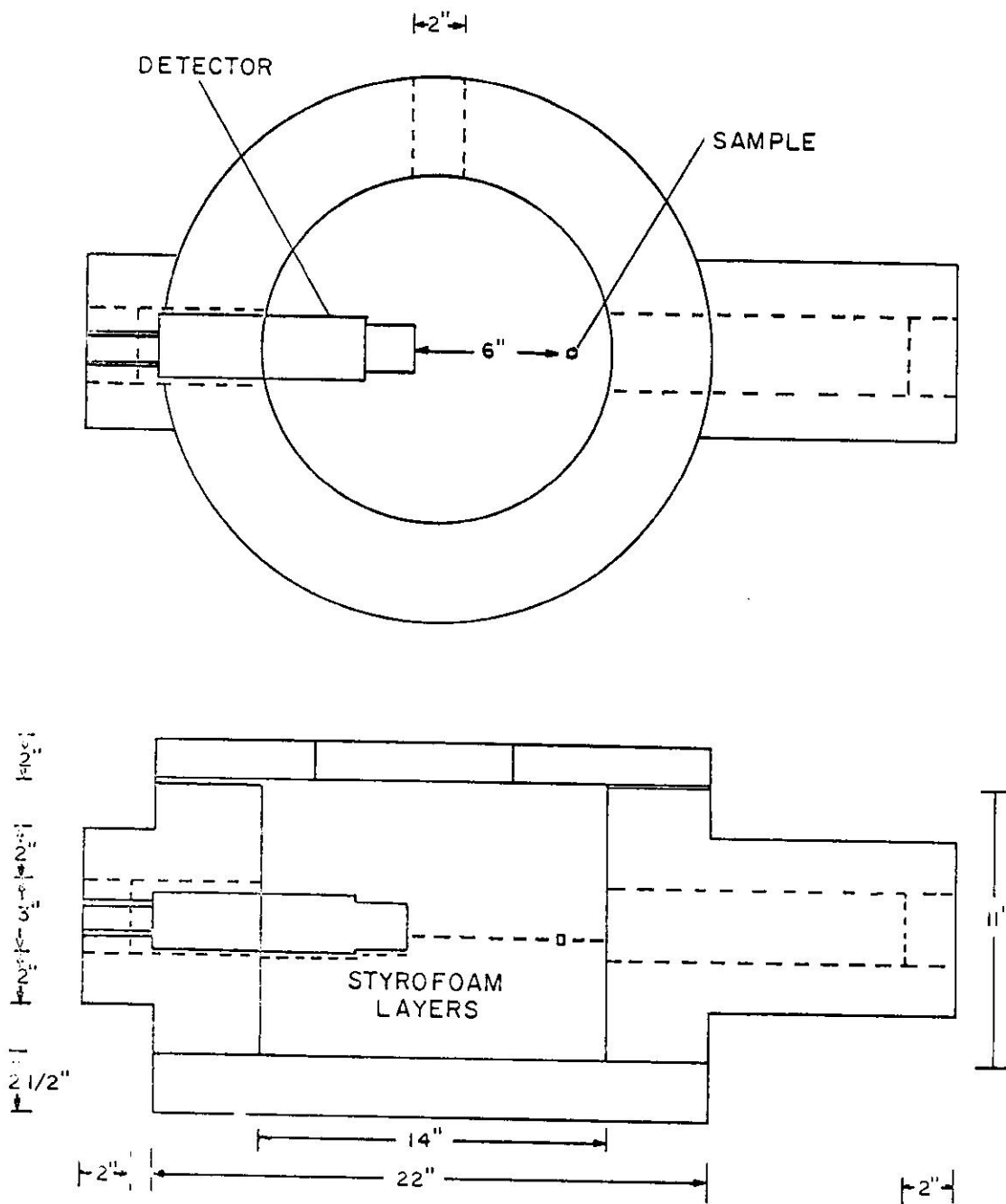
Fig. 5

SAMPLE COLLECTOR AND BOTTLE

Fig. 6

I ml OF SEA WATER

IRRADIATION TIME : 3 SEC.
DECAY TIME : 30 SEC.
COUNTING TIME : 80 SEC.

Na^{24} (1.37 MeV)

Cl^{38} (1.66 MeV)

Cl^{38} (2.10 MeV)

Fig. 7

CHANNEL NUMBER

NUMBER OF COUNTS
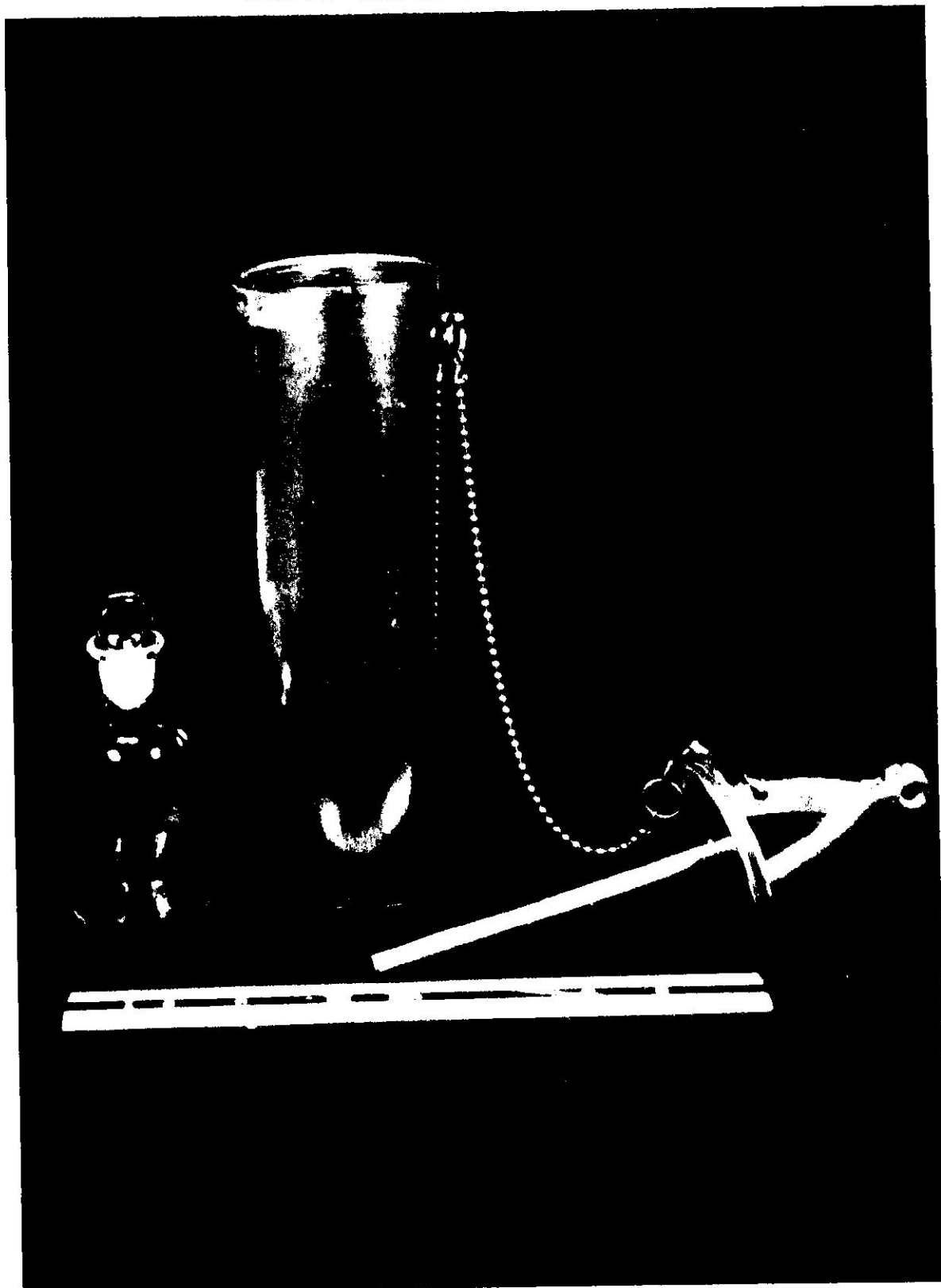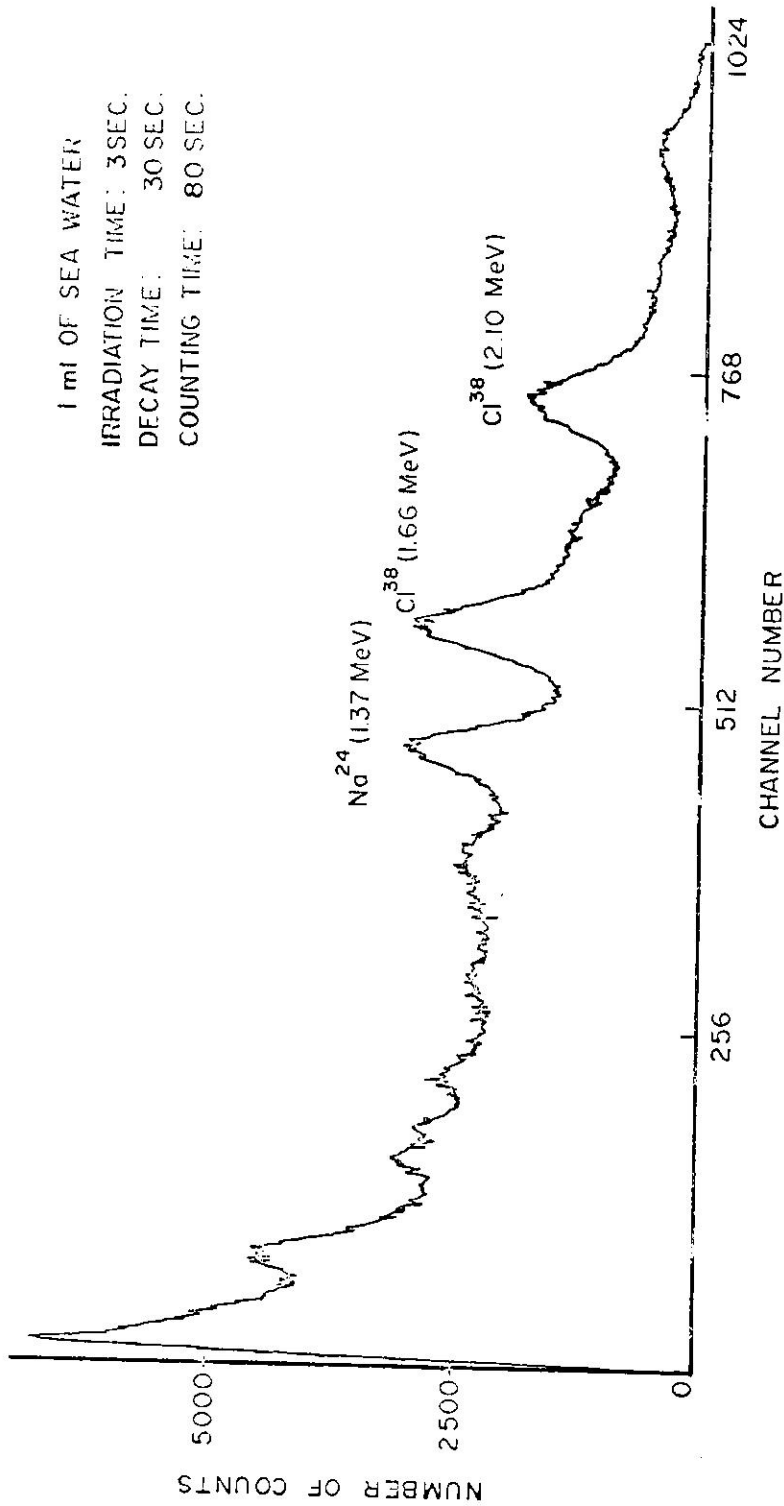
papers before and after filtration.

Sequence of Analysis

The filter papers were folded and placed in cylindrical polyethylene vials, one inch long and 1/2 inch in diameter where they were irradiated for 3 seconds together with vials containing aluminum standards. The aluminum standards consisted of small pieces of aluminum foil which were accurately weighed. Both the samples and standards were located at the bottom of the vials in order to sustain the same geometry relationship with the detector. They were analyzed in alternate order for 40 sec live time, for a period of time which did not exceed five minutes.

The multichannel analyzer had been calibrated to read from 0 to 3 Mev in the four sections of the memory using an aluminum standard. The aluminum photopeak was identified and integrated from 6 channels before to 6 channels after the peak channel. The result of these integrals were then plotted on semi-log paper.

Since the decay times have to be equal in order to make equation 16 valid, the ratio of the integrals have to be taken at a fixed decay time. For this work the value of the integrals 2 min. after irradiation was taken. By substitution in equation 16 the weight of aluminum in the samples was determined.

Results

The results of this work show that the amount of aluminum present in the particulate matter suspended in sea water can be measured using instrumental neutron activation analysis. A summary of the results is presented in Table 1.

TABLE 1: SUMMARY OF RESULTS

Sample Number	Location of Sampling Stations	Depth	Weight of Particulate Matter	Volume of Water	Weight of Aluminum	Concentration of Aluminum	Weight of Particulate matter per liter of water	Weight of Al per gram of particulate matter
1	20 meters windward side of the outfall	1 meter	2.4 mg.	287 ml.	0.20 mg.	0.71 mg/l	8.4 mg/l	85 mg/gm
2	Origin of outfall	1 "	3.2 "	272 "	0.29 "	1.0 "	11.8 "	89 "
3	50 meters from the outfall	1 "	4.1 "	285 "	0.30 "	1.0 "	14.3 "	72 "
4	100 meters from the outfall	1 "	7.4 "	280 "	0.29 "	1.0 "	26.4 "	40 "
5	200 meters from the outfall	1 "	2.1 "	286 "	0.20 "	0.68 "	7.3 "	93 "
6	250 meters from the outfall	1 "	3.7 "	292 "	0.27 "	0.94 "	12.7 "	74 "
7	400 meters from the outfall	1 "	3.4 "	292 "	0.36 "	1.2 "	11.6 "	100 "
8	100 meters from mouth of Yaguez River	1 "	18.6 "	292 "	1.7 "	5.9 "	63.7 "	92 "
9	20 meters windward side of the outfall	5 "	13.4 "	297 "	0.75 "	2.5 "	45.1 "	56 "
10	Origin of the outfall	5 "	19.2 "	283 "	1.0 "	3.6 "	67.9 "	54 "
11	500 meters from the outfall	4 "	36.1 "	292 "	2.7 "	9.4 "	123.5 "	76 "
12	100 meters from the mouth of the Yaguez River	4 "	68.7 "	200 "	4.4 "	22 "	343.5 "	64 "

A typical representation of the Al^{28} photopeaks (1.78 MeV) obtained from the activation of the particulate matter, as compared to that obtained from the aluminum standard is illustrated in Fig. 8. The first and third correspond to the suspended sediment sample while the second and fourth were obtained from the aluminum standard.

In Fig. 9 the integral over the Al^{28} peak for the sample and the standard is plotted as a function of the decay time.

Similar graphs were obtained from all the samples of particulate matter and the amount of aluminum was found to be dependent on location and depth. The area where the samples were collected, and the collection points, are shown in Fig. 10.

In Fig. 11 the concentration of aluminum is plotted for different locations and depths. The shape of the curve for samples taken at a depth of 1 meter shows that the waste discharged at the sewage outfall and in particular the suspended matter carried by the Yaguez River contain large amounts of aluminum. Curve segments which are also plotted in Fig. 10 show that the aluminum concentration increases with depth. This effect is more significant in the vicinity of the Yaguez River where rapid settling of the sediment carried by the river is occurring.

Discussion

The fact that sodium and chlorine exist in sea water in high concentrations constitutes the first foreseeable problem when analyzing marine samples. When the samples are activated the masking effect of Na^{24} and Cl^{38} does not allow the detection of the minor constituents. This problem was successfully overcome by filtering the water samples with

AL^{28} PEAKS FROM SAMPLE AND STANDARD

Fig. 8

CHANNEL NO.

NUMBER OF COUNTS

SAMPLE

STANDARD

STANDARD

SAMPLE

DECAY CURVES FOR SAMPLE AND STANDARD

Fig. 9

APPROXIMATE LOCATION OF SAMPLING STATIONS

NORTH

MAYAGUEZ
BAY

Yaguez
River

City of
MAYAGUEZ

Location of
sewage outfall

Highway
No. 2

SCALE: 1:15000

Fig. 10

ALUMINUM CONCENTRATION VS. LOCATION
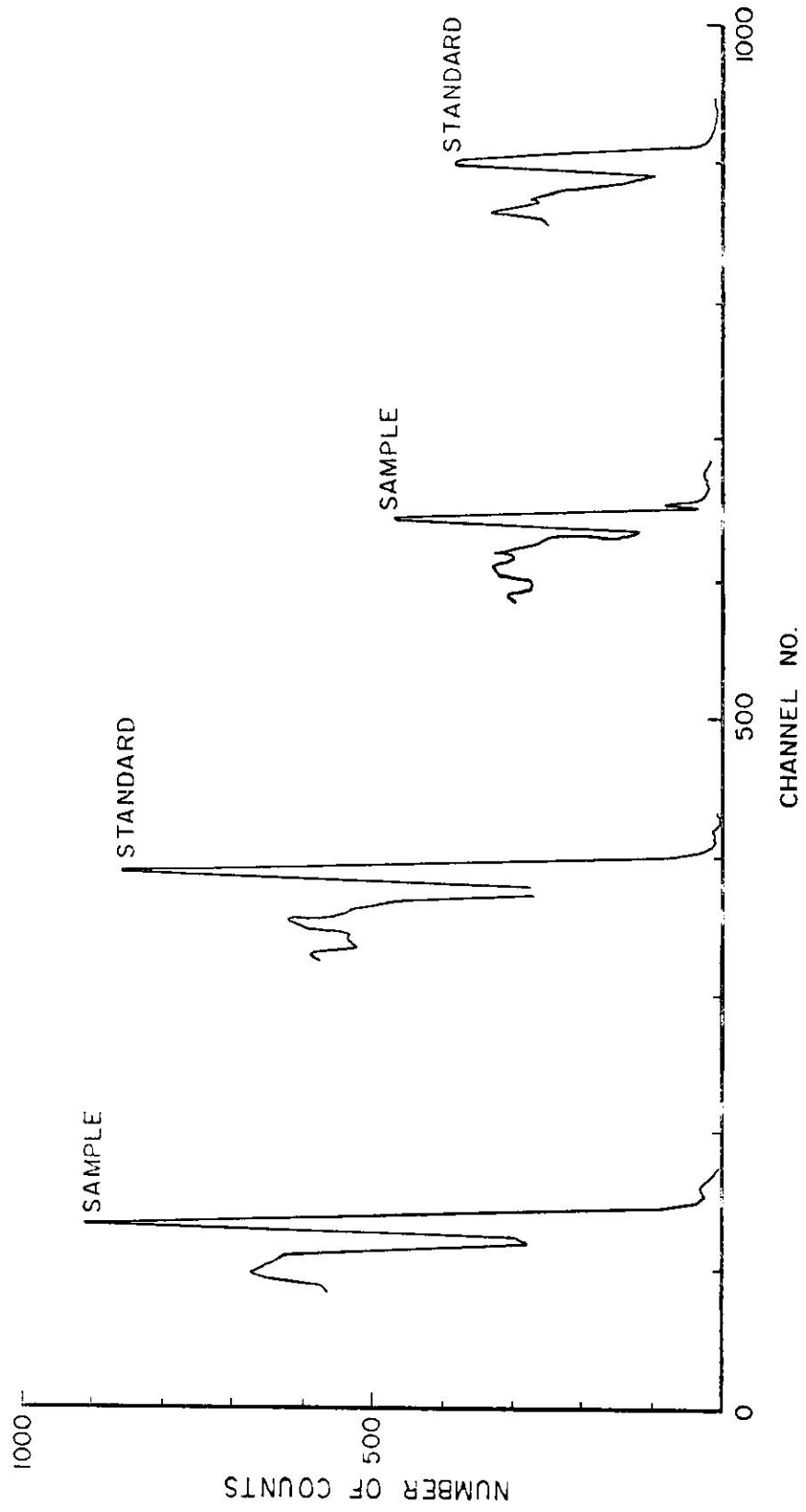
Fig. 11

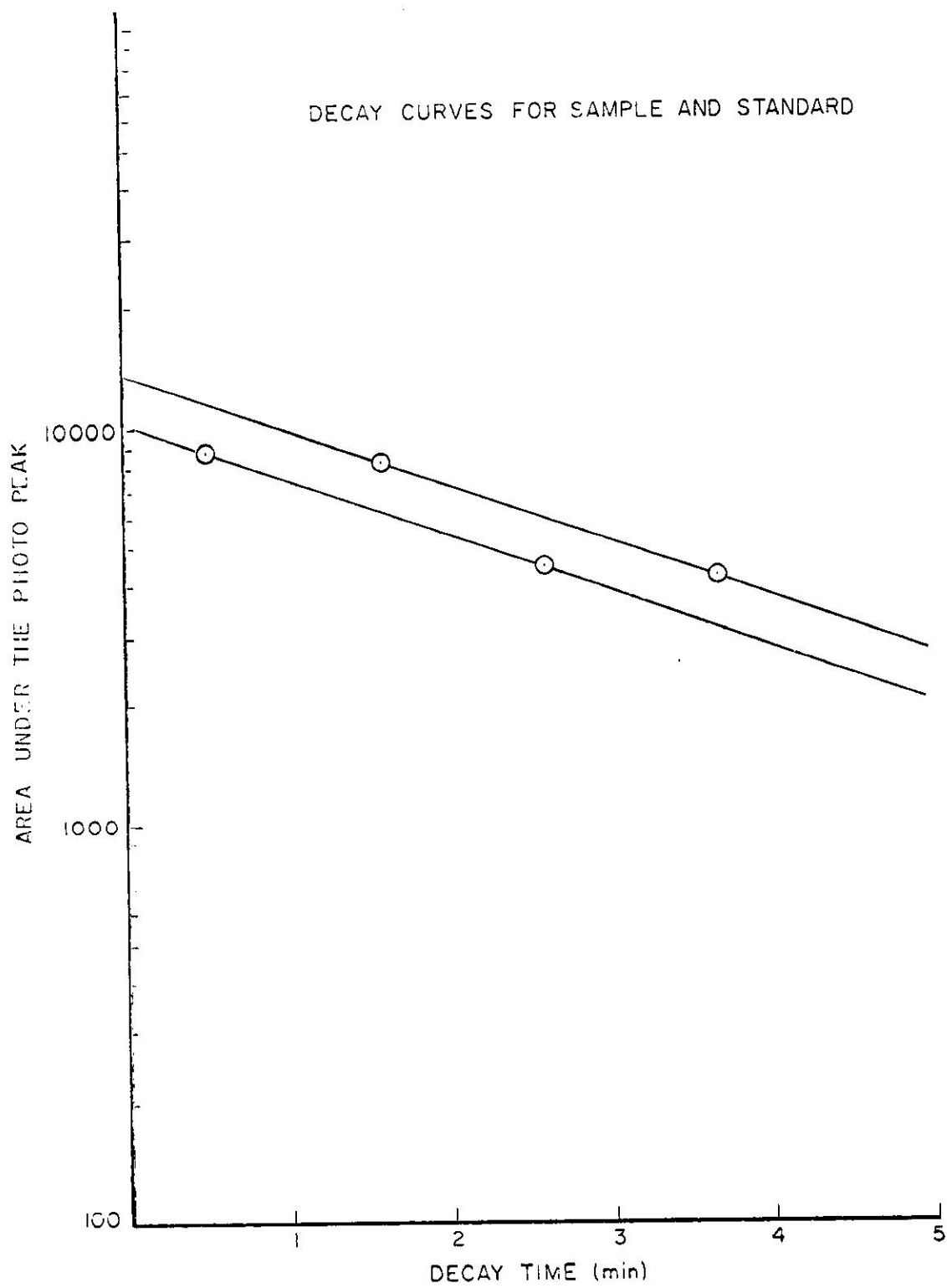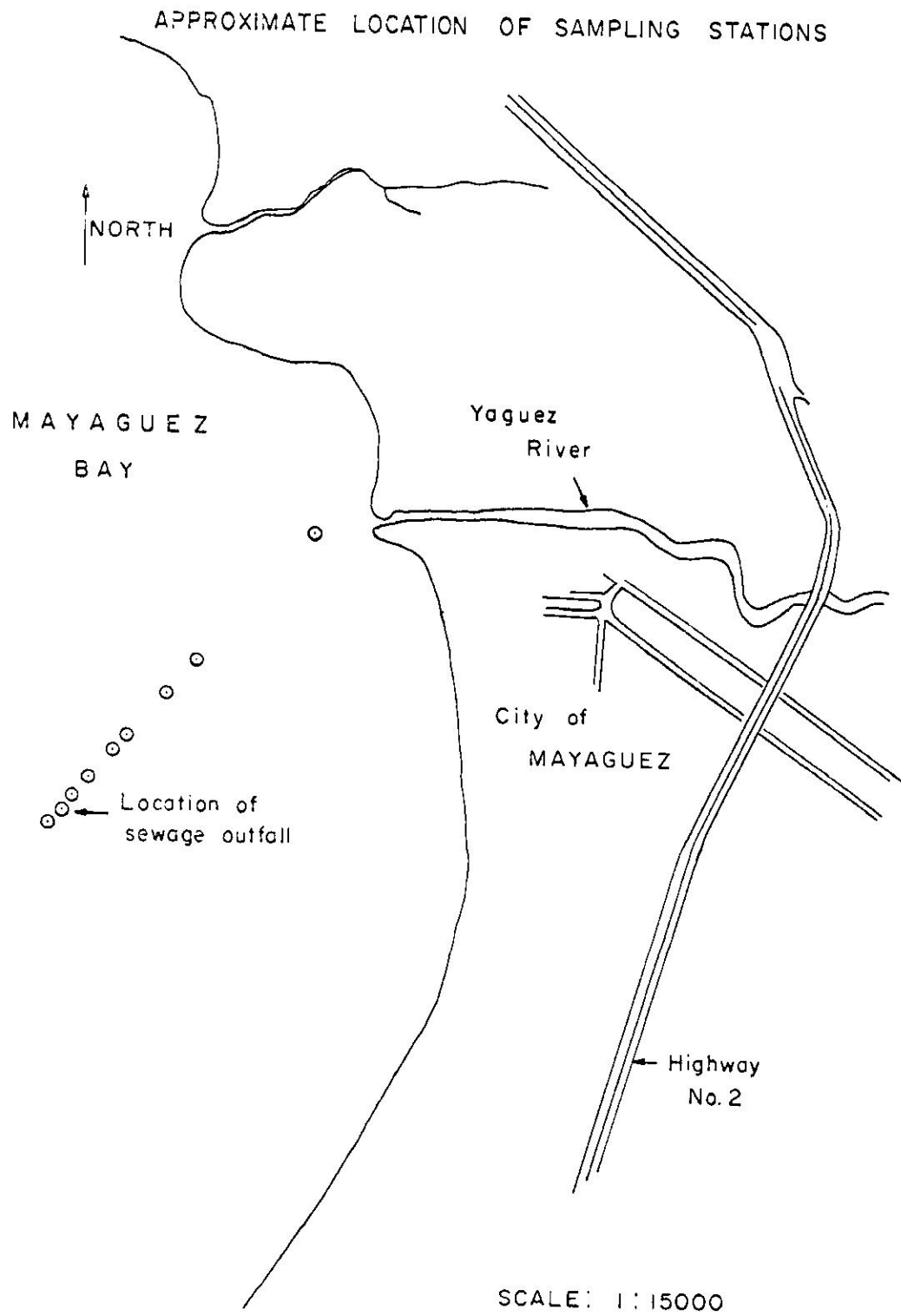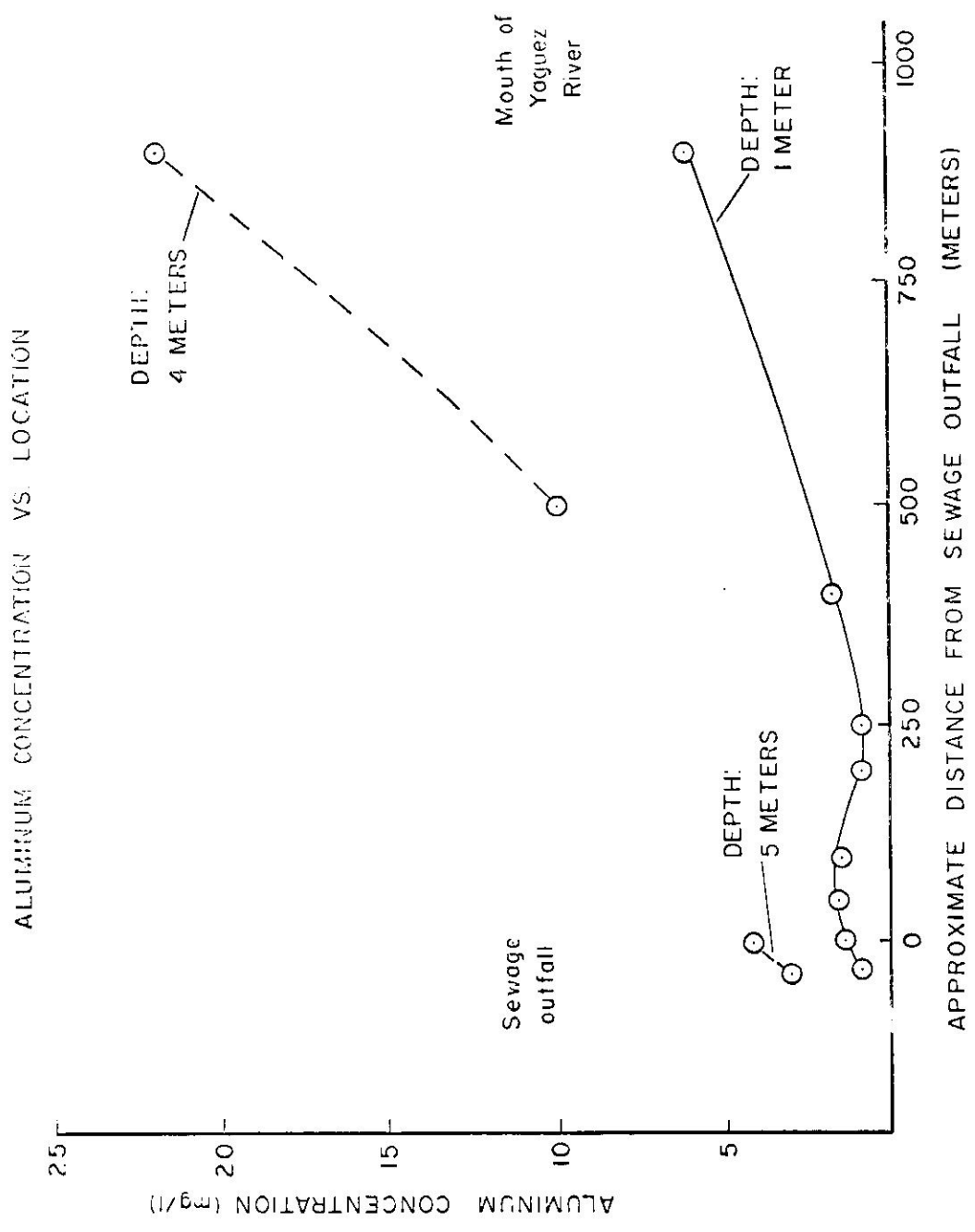0.45 μ millipore filter paper. During filtration the particulate matter which is not in solution is retained in the filter paper while the large amounts of sodium and chlorine which are in solution go through. This reduced to a negligible level the interference of these two major constituents, thus making possible the detection of small amounts of other active products.

When using standards to perform quantitative analysis by neutron activation, one of the most important considerations is the fact that the shape and the size of the standard and of the sample should be similar. For very small samples the variations in efficiency due to the difference in shape and size of the samples and standards is only significant when they are located close to the detector. When they are placed at a distance of 10 cm or more the difference in efficiency becomes negligible[12].

Another important consideration in the use of instrumental neutron activation analysis is the choice of the irradiation and decay times. These should be chosen so that the ratio of the activity of the desired radioisotope, to the activity of the radioisotope which would create the greatest interference, is a maximum. Calculations done to optimize these two parameters for this experiment show that for measuring aluminum in the presence of sodium the shortest possible times of irradiation and decay should be used. If the radioisotope which is going to be produced has a short half-life like for example Al^{28} ($T_{1/2} = 2.3$ min.) another consideration in the choice of the irradiation time should be to produce enough Al^{28} to be able to measure its activity

during at least two half-lives.

A considerable effort has been devoted to the study of those reactions that take place at the river-ocean interface which modify the composition of the near shore waters. If the sediment coming from the rivers can be identified at the ocean-river interface, the pattern of sedimentation at the river mouth can be determined. In order to identify the sediment which is coming from the river, a tracer must be used.

The fact that the concentration of aluminum in the suspended solids was found to increase in the vicinity of the Yaguez River, suggests the possibility that aluminum may be used as a tracer for the river sediments. Since aluminum is an integral part of the river sediment, its rate of settling should be the same as that of the river sediment.

One of the advantages of knowing the pattern of distribution and settling rate of river sediments is that the blocking effect which is developed at the mouth of the river could be predicted, and preventative measures could be developed. It is of fundamental importance that the river waters are allowed to flow freely into the ocean, since the limitation of the water flow not only endangers its quality but may cause flooding during seasons of high precipitation.

The technique which has been developed here is designed to use aluminum as a naturally occurring tracer. In situations where aluminum can not be used as the tracer other tracers can be developed by changing the times of irradiation and decay of the samples.

A few improvements should be made to the water sampling technique. Since for this research project the greatest effort was devoted

to the laboratory analysis, the determination of the location where the sample was collected was done only visually. A more precise method would be used if a study of the ocean-river interface were to be conducted. Also in order not to disturb the sample at the point of collection, a sample collector which could be opened and closed at the desired depth should be used.

CONCLUSIONS

From the results of this work the following conclusions can be drawn:

1. By irradiating 1 ml of sea water and analyzing it after decay times up to 30 sec only sodium and chlorine can be detected.

2. By filtering the water samples using 0.45 μ millipore filter paper and analyzing the filter paper by neutron activation, the sodium and chlorine interference is reduced and the amount of aluminum present in the particulate matter suspended in the water can be measured.

3. The concentration of aluminum in the Mayaguez Bay area which was studied, was as much as one hundred times greater than the value for standard sea water.

4. The concentration of aluminum and the weight of particulate matter per liter of water, increases in the vicinity of the sewage outfall and near the mouth of the Yaguez River.

5. The concentration of aluminum and the amount of particulate matter suspended in the water also increase with depth.

6. The method and techniques developed in this work can be used to trace the distribution and settling rate of the particulate matter contributed by certain river waters.

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