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PUERTO RICO NUCLEAR CENTER

ACTIVATION ANALYSIS AS A METHOD FOR

TRACING SUSPENDED SEDIMENTS,

Braulio Mejia Avilés and Knud B. Pedersen

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ACTIVATION ANALYSIS AS A METHOD

FOR TRACING SUSPENDED SEDIMENTS

by

Braulio Mejfa avilés

ane

Knud Borge Pedersen

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assTRACT

Neutron Activation Analysis was applied to the problen of

jedinentation in the Mayaguez Bay. The method vas chosen as an attempt to eliminate the cunbersone chenical methods normally ?employed, and because it offered the opportunity to increase the sensitivity greatly.

24 ana ci? were elininated by

?The masking effect of Na

filtering the samples through 0.45 u millipore filter paper. The

papers were alloved to dry in @ closed hood to nininize airbore

contanination and dust fron entering the samples. The weight of the particulate matter in the filter paper vas 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 Nal (11) crystal, The samples and standards vere counted in altemate order for 40 sec Live tine, for a period which did not exceed 5 nin. Aluminum vas found to be

present in all the

jeples in different concentrations.

?The results denonstrated that aluminum may be used for tracing

the sediments contributed by river waters, offering the opportunity of

Jetermining their distribution pattern and settling rate.

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LIST oF FIGURES

Diagran of the Detector Shield,

Sample Collector and Bottle.

Spectrun From 1 ml Seawater.

ae

Peaks From Sample and Standard,

Decay Curves for Sample and Standard...

Approximate Locations of Sampling Station....+-.++++

Alusinus Concentrations v. Locations.

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23

4

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INTRODUCTION

Neny new techniques have developed almost as by-products of suclear Fever, Activation Analysis is one of then, ?Activation? by sone type of ?uclear reaction is used te produce a radioisotope fron the elesent to be Gsterined. There are two major advantages in using activation analysie: first, since the instrumentation available novadeys allove the detection

and measurement of very small anounts of radioactivity, this proves to be

* Ory sensitive sethod, second, since the radioisotope formed decays

with its om of

acteristic radiations and half-life, it is feasible to

sake activation analysis very specific. Besides its high sensitivity,

activation analysis is a fast and econonical sethod of analys

Of al the possible nuclear reactions, the neutron reactions heve

been most often used. In the analysis of complex mixtures of nuclices

the major constituents with high neutron eros.

sections mask the spectra

of other trace activation products. Most of the methods that are used

$9 analyze complex mixtures by neutron activation enploy suitable chemical

separations to elininate the interfering induced activities. If they are

done before irradiation, sone of the interfering activities would be

intensified or new ones vould be introduced. in general these chenical

erations are time consuming and therefore, if done after irradiation,

ae not practical when analyzing for short-lived isotopes.

When analyzing for short-lived isoton

entirely instrumental

methods rust be used. By analyzing the sample iemediately after ir-

Fadiation, very small anounts of these short-lived isotopes can be

reasured. In thin vork @ technique w

veloped to measure snail

amounts of aluninun present in sea water samples.

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[REVIEW OF LITERATURE

Increasing interest has been shown in purely instrumental

ethods of neutron activation analysis, and a nusher of studies not

Anvolving chenical separations have been reported.

(1D jap published a review of some recently developed

v.P. Guinn

jnserumental methods and techniques which are now in use, Sone of these ethods employ pneunatic tubes to analyze the samples as short as tvo

seconds after irradiation.

fo. v. Anders(3*4) as applied the concept of gamma-ray difference

apoctrun to suppress the longer-Lived components in the gamma spectra of activated samples, The method consists in analysing the samples shortly after irradiation and also after the short-Lived {aotopes have decayed, by substracting the second spectrum from the first, the effect of the tonger-Lived isotopes is alnost entirely renoved from the gamma-ray spectrin.

?

Dik, Robertson, L.A. Rencitelli and RW, Perkins?) used neutron activation and direct counting techniques to measure concentrations of

punerous trace elenents in sea vater, marine orgeniens and pelagic sedi-

mont

Rit, Caldwet) and W.R, Mitie, Je. 6 have used rapié neutron
activation techniques for quantitative determination of Silicon, Aluminun
?and Yagnesivm in rocks. The accuracy and reproducibility of the snaly-
es and interferences from other elenent are given,

nit. creene?? has reviewed the us

of activable stable isotopes

with various types of activation analysis for tracing, Advantages and

disadvantages of the nethod are given, and several exanples of applications

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in medicine, pollution studies and hydrology are described.

P. Marrenoes (20?

has described the use of radioisetopes in

tracing the effluent disversion at a proposed site for sevaze dia

posal, Tracer injections at different sites permit a selection of

the nest economical site according to the required purification of

the sevage.

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nen a stable nuclige captures neutrons and forms a radioactive

product which has relatively low absorption cross section, the rate of

change of the nunber of atoms of the radicactive product will be given,

ane, Nao?

at

= ance) a

where

N(t) = Number of radioactive atons

= Avogadro's number

= Meight of the stable nuclide

@ = Keutron activation cross section

A= Atomic mass number of stable nuclide

@ = Neutron flux

A Decay constant of the radioactive product

multiplying both sides of equation 4 bye

at Nee ae ?

ay Bh QM sel nce) @

eee. et @

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se Jews Mt

Then integrating, and assuming (0) = 0

Hor? net

aCe f eee at ?)

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where AN(t) is the activity of the radioactive product. The activity

after irradiation tine t and a decay tine is given by

Niyee

pet (eye) = BREE GQ @

and the count rate will be,

Gok. (yt) © det CE) o

where © is the

ficiency of the detector. For # point source on

the axis of a cylindrical scintillator as in Fig. 1 it is given by,

cs Sone eo ao

where Go is the solid angle subtended by the crystal measured from

the point source, and fin the total absorption coefficient.

Quentivative Analysis

In order to perform quantitative analysis of any sample

using activation analysis, we can measure the flux, cross section and

efficiency of our systen, or compare our sanples with known sanples of

standards. tn order to conpare the activity of the sanple with that

of a standard, they must have been exposed to the sane neutron energy

spectrum and neutron flux, and have sinflar count rates, thus producing

essentially the sane detector dead tine, To obtain the sane detector

efficiency, the geonetriex sust also be equal.

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WLUSTRATION FOR CALCULATING EFFICIENCY

crstet t

Fig. 2

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by irradiating the canple and atandard for the sane tine

ani essentiallyin the sane place we can assure the saee value of the

flux and the sane nevtron energy spectrum. Sy choosing the weight of

the standards so that their count ra

5 will be sinilar to that of the

sarples, similar dead tines can be obtained. If the standard and the

sample are located in the sane place relative to the detector,

and

having essentially the sane dead tine the same efficiency can be assuned.

Taking the ratio of the count rate of the sample to that

of the standard ve get,

tens = Eta woot ne ey a

stp SMa Ksrmetca - e7Myg AV A

Therefore, if the decay tine is the sane

where Wir, is the weight of the standard.

THE EXPERINE!

Reser

-iption of Electronic fquipment

1, Scintiitation Detectors

ay

2p

as

4.2" x 2" sodium iodide crystal activated with thalliun vas

sed for the detection of ganna ra

Te was coupled to a 10 stage,

---Page Break---

photomultiplier tube which was operated at 935 volts.

2, Preamplifier

ne purpose of the preasplifier is to inerease the signal amplitude froe the detector and provide inpedance transformation; that is, i transforms the voltage which is developed across the snalt ca pacity at the input of the preanplifier into an approxinately equal voltage across the high capacity output cable. Canberra Industries Nogel 805 Seintillation Preamplifier was used for these experinents,

Ie is 9 charge

jensitive, all

Jilicon transistor device vhich integrates

cue charge output signals from seintillation/photonultiplier detectors,

for presentation to the pulse shaping main amplifier,

3. Amplifier

?The function of this instrument is to increase further the

?sinnal amplitude from the preamplifier. The gain can be adjusted to

cbtain the desired energy scale in the analyzer. C.1, Model 810 double

delay anplifier was used for this experiment, It accepts the pre~

avplified signals fron proportional counters, scintillation

or seniconductor detectors. It yields in turn output signals suitable

for single channel or rultichannel analysis, and for leading cdre oF

ing timing.

4 Wigh Voltage D.C. Supply

hia instrument transforms the 115 Volt, 60 6.7.5. A.C.

voltage to high D.C. Voltage to be used by the photomultiplier tube,

?the one used for this experinent vas @ Fluke Model 409 A, whose ouput

ranges to 1,500 volts D.C. with Less than .0U2% ripple and Ina. output.

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5. in and Power Supply

Ortee Model 401 A Modular System Bin was used to provide

power for the amplifier. It can provile plus or minus 12, or 24

volts D.C. and 115 A.C, delivered to ansizned rodule connector pins.

6. Multichannel Analyzer

Tue Multichannel Anelyzer consists of the folloving elenents:

(2) the analog to digital converter that associates each input signal

with a specific anplitude channel, (2) the menory, oF data

vhich contains the infornation of te nurber of pulses in each channel,

and (3) the osciMoscope, typewriter, and curve plotter whicl: provide

For the display of the data which is stored in the menory. The system

for this experivent was a fuclear Data series 2201 aysten analyzer, which

is a completely eodular multichannel analyzs

A block éfajran of the electronic equipment is shown in Fig. 2.

ALL the irradiations were gone at the T.R.N.C. research reactor at a

power level of 1.

Transfer Syaten

1, Description of the systen

The syster consists of @ pipe through which the ?eabbie",
(tie velicle wiwre the samples are placed), travels to and fro the
reactor, The tire of irradiation can he adjusted frce one second up
to thirty minutes, hen the rabhit dispatch button fs pressed, vacuum
is applied at the reactor end of the pipe, thus pulling the rabbit in
tovaré the reactor. when vacuum is applied instead at che other ené

of the pipe the rabbit is made to return.

2, Modification Done to the System

Fig. 3 illustrates the modification done to the pneuatic

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MODIFICATIONS DONE TO THE PNEUMATIC system

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To tne Reactor

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Short Pipe

Connection

Detector Shiels

Fig 3

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trmafer systes. ?hen the system was set on "rabbit return" the ?yacuun applied above the receiving bex to make the rabbit return from the reactor also pulled the rabbit back after it had passed the vacuur connection, tix nade the rabbit loose ainost all of its speed making it impossible to reach the end of the pipe. In order to renove the vacuum effect a butterfly valve was installed in such @ eanner that it was activated by the rabbit as it approached the vacuum connection. by installing a corved pipe below the receiving box the rabbit was trans~ ferred fron the reactor directly inte the detector shield. A short pipe inten was placed inside the receiving box alloved the rabbit to by-pass the box.

?the detector shield used for this experiment is shown in Fig. 4.

te was wade from solid lead in the form of a hollow cylinder, The

circular valls are four inches thick and have three circular

cavities, tvo for the detectors and one for the rabbit tube. The to

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 tvo inches thick and tve lead plugs of the same thickness

are located behind each detector.

inner walls of the shield vere covered with .027" of cadnive

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 aysten, it had

to be sealed air tight, so chat the vacuun would effectively pull the

rabbit from the reactor.

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wo styrofoam layers vere placed inside the shield to provide suport for the detectors, The detector was enbedded in the top layer thus reeaining in a fixed position.

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

D. Sample Collection and Preparation

Sen water samples were collected usiny. the sanple collector shoun in Fig. 6. Clase bottles vere previously washed vith 4N Nitric Acid and distilled water, ty holding the sanple collector at the desired depth a bottle placed in the semple collector will be completely filled

with Use surrounding water, Sanples vere collected at different locations

ond at different depths in the vicinity of « sewage outfall in the Navaquee Bay.

Since the purpose of this project was to develop @ technique,

so that the samples could be irradiated and analyzed without chesical separations, 1 wl. sanples of the vater vere irradiated, using anal polyethylene vials, and were then directly analyzed. The water semples vere irradiated for three seconds and analyzed after thirty seconds of gecay. Only Sodium Chloride could be detected as can be seen in Fig. 7 here one such spectrum is shorn.

The renainder of the sea vater sanples were filtered, using 0.45 4s millipore filter paper, measuring one inch in diameter. The filter papers vere allowed to dry in a closed hood to wininize airborne

contamination and cust from entering che samples. The weight of the

particulate matter in the filter paper was determined by weighing the

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DIAGRAM OF THE DETECTOR SHIcLO

DETECTOR

STYROFOAM

LL | LAYERS.

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vas Jo my

?ona

u3ennN TaNNeHO

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papers vefore and after filtration.

Sen

nee of Analysis

?The filter papers were Folded and placed in cylingrical

polyethylene vials, one inch lonz and 1/2 inch in dianeter where they

vere irradiated for 3 seconds together vith vials containing alurinun

standards, The alurinun standards consisted of snall pieces of

alusinun foil whieh were accurately weighed. Both th

samples ang

standards were located at the botton of the vials in order to sustain

the vane geenetry relationship with the detector. They were analyzed

in alternate order for 40 sec live tire, for a period of tine which ete

rot exceed five einutes.

?The multichannel analyzer had been calibrated to read from

0 to 3 Yev in the four sections of the menory using an aluninun standard.

identified and integrated fron 6 channels

?The aluninur: photopeak x:

before to channels after the peak channel. The result of these in-

tegrals were then plotted on semi-Log paper.

Since the decay tines have co 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 nin, after irradiation was taken, hv substitution in equation 16 the weight of aluminum in the samples was determinee.

Results

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

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A typical representation of the A1"® photopeaks (1.78 rev)
obtained from the activation of the particulate matter, ax compared to
that obteined fron the aluninur standard is illustrated in Pig. 8. The

First and third corresnond £0 the suspended sediment sample while the

second and fourth vere obtained from the aluninun standard.

In Pig. 9 the integral over the A1® peak for the sample and
the standard is plotted as a funetion of the decay tine.

Similar graphs were obtained fron all the sarples of particu-
Late matter and the anount of aluminum was found to be dependent on
location and depth. The area where the samples were collected, and the
collection points, are show in Fig. 10.

In Fig. 11 the concentration of aluninun is plotted for
Gifferent Locations and depths, The shape of the curve for sanples taken
at a depth of 1 meter shous that the waste discharged at the sevage out
fall and in particular the suspended ratter cerried by the Yaguez River
contain large arounts of aluminur. Curve segments vhich are also plotted
in Pig. 10 show thet the alusinun concentration inereases with depth.

This effect is nore significant in the vicinity of the Yaguez River where

rapid settling of th sediment carried by the river is occurring.

the fact that soiur and cilorine exist in sea water in high

concentrations constitutes the first foresecable problen viren analyzing

narine sanples. when the sanples are activated the wasking effect of

xa ane ca?# goog not allow the detection of the =

?This problen was successfully overcone by filtering the water samples with

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(ON 73NNVHO

a

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2

[DECAY CURVES FOR SAMPLE ANO STANDARD

10000|-

ooo}

yoo

t 2 s = 3

DECAY TIME (min)

Fig. 9

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APPROXIMATE LOCATION OF SAMPLING STATIONS

yvonra

Yoouez

River

MAYAGUEZ

Bay

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2 Locotion of

sewage outfall

Highwoy

No.2

SCALE: 1115000

Pig. 10

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OG n wfilipore filter paper. During fileration the particulate watter
which fs not in solution is retained in the Filter paper vile the Large
?Srousts of sodium and chlorine which are in solution go through. thie
Seisced to @ negligible Level the interference of these two eajor con
?titwents, thus walking possible the detection of cnall amounts of other
active products.
?ten weing standards to perform quantitative analysis by

?eutron activation, one of the sost inportant considerations is the fact
?hat the shape and the size of the standard and of the sample should be
Sinttar, Tor very snolt samples the variations in efficiency due to the
Sitference in shape and size of the samples and standards és only aige

nificant when el

Y are located close to the detector. \hen they are
laced of a distauce of 10 em or wore the éfference in efficiency

becomes negligibie(!2),

Another important consideration in the use of instrumental

evtten activation analvsin Is the choice of ee irradiation ané decay

Sines~ These should be chosen so that the ratio of the activity of the

tenired radioisotope, to the activity of the radioisotope vhich would

Create the greatest interference, is a nexium. Calevlations done to

ontinize these two paraseters for this experiment shov that for

nessuring elusinun in the presence of sodiun the shortest po

ible tines

of irradiation and decay should be us

IE the radioisotope which is

Reing to be produced has a short half-life like for example 4122

(xa * 2-3 min.) another consideration in the choice of the irradiation

ise should be 0 produce enough Al°® to be able co measure its activity

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2%

during at Least two balf-Lives.

A considerable effort has been devoted to the study of these reactions that take place at the riverocean interface wich modify the couposition of the near shore waters. If the sedinent coming frow the rivers can be identitied at the ecean-river interface, the pattern of sedimentation at the river nouth ean be determined, In order to igenti-fy the sedinent which is coning fron the river, a tracer must be used,

?the fact that the concentration of alurinus in the suspended solide waa founé to inerease in the vicinity of the Yaguer River, sus

nents the poseibility that aluninun may be used ae a tracer for the

river sediments, Since aluninun is an interral part of the river sedi

pent, ite rate of settling should be the sane as that of the river

one of the advantay

1s of knowing the pattern of distribution

and settling, tate of river sediments is that the blocking effect which

is developed at the routh of the river could be predicted, and pre~

ventative rei

sures could be developed. It is of fundarental irportance

chat the river waters are alloved to flev freely into the ocean, since

the linitation of the vater flow not only endangers its quality but ney

caune floeding during seasons of high precipitation.

?The technique whieh has been developed liere i

igned to use

aluninun as a nat

rally occurring tracer, In situations where aluminun

can not be used ss the tracer other tracers can be developed by chansinn

the tines of irradiation and decay of the samples.

A few inprovenents should be made to the water sampling tech

nique, Since for this rescareh project the greatest effort was devoted

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to the lohoratory analysis, the deterrination of the location where the

Sample vas collected vas done only vinually. A more precise wethod

Would be used if a study of the oce

w-tiver interface were to be con

ducted. 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.

coxcnustoss

Fron the reaults of this work the folloving concluaions can he

dram:

by irradiating 1 rl of sea water and analyzing it after decay

Himes up to 30 see only sodium and chlorine can be detected.

2. by Cltering the vater samples using 0.45 u nillipore filter

paper and analyzing the filter paper by neutron activation, the sodiun

and chlorine interference is reduced and the anount of alusinum present

in the particulate matter suspended in the water can be measured.

3. The concentration of aluminus in the Mayaguez Bay area which

was studied, was as much as one hundred tines greater than the value

for standard sea water.

?Tue concentration of alurinum and the veight 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 aluminur and the anount of particulate

hatter suspended in the water also inereare with depth.

4. The method and teclmiques developed in this work can be used

to trace the distribution and settling rate of the particulate catter

contributed by certain river vaters.

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