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## An Evaluation of a Stable Isotope of Dysprosium for Labeling and Tracing Sedimentary Particles

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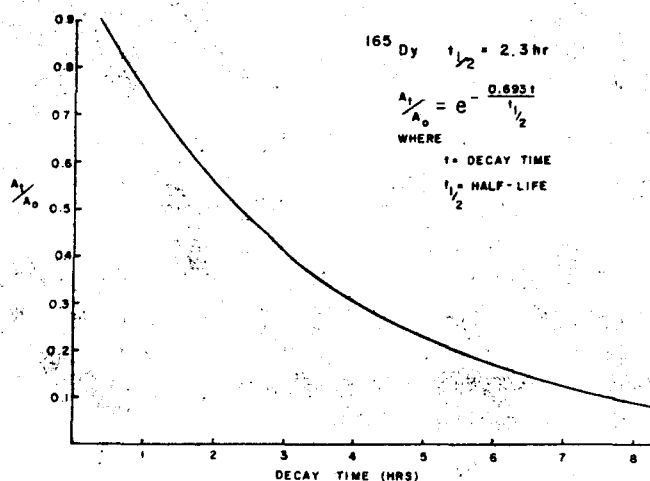
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# AN EVALUATION OF A STABLE ISOTOPE OF DYSPROSIUM FOR LABELING AND TRACING SEDIMENTARY PARTICLES



Charles Glenn Boone

AN EVALUATION OF A STABLE ISOTOPE OF  
DYSPROSIUM FOR LABELING AND TRACING  
SEDIMENTARY PARTICLES

by

Charles Glenn Boone

A THESIS

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Old Dominion University, Norfolk, Virginia  
in partial fulfillment of the requirements

for the degree of

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Committee

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## ABSTRACT

An evaluation of the application of dysprosium oxide as a tracer coating to surface label sedimentary particles is made. Physical and chemical properties of the labeled sand are studied. Effective diameters and settling properties of the tracer sand are compared with unlabeled sand. A total of 8.51 kg of labeled sand was placed on the beach at Camp Pendleton. Dispersal patterns obtained 1 and 4 hours after tracer injection indicate a general downcurrent mean tracer migration rate of 0.21 cm/sec (0.42ft/~~min~~<sup>sec</sup>). An absolute detection limit of 4 or 5 parts per 100,000 and a real detection limit, after field dilution, of 1 part per 10,000 was obtained. Discussion of the future application of the stable isotope method is presented.

## TABLE OF CONTENTS

	Page
INTRODUCTION .....	1
General Statement .....	1
Previous Studies .....	2
Radioisotope Studies .....	2
Fluorescent Studies .....	4
Stable Isotope Studies .....	5
Research Approach .....	8
Limitations .....	10
METHODS .....	12
Laboratory Methods .....	12
Sand Preparation .....	12
Labeling Procedures .....	12
Evaluation of Effective Diameter and Settling Properties of Tracer Sand .....	13
Abrasion and Leaching Studies .....	13
Preparation of Samples for Activation .....	14
Evaluation of Stripping Procedures .....	17
Activation Analysis of Field Samples .....	18
Field Methods and Procedures .....	21
Test Site .....	21
Sample Grid .....	21
Injection of Tracer and Recovery of Samples .....	26
Measurement of Physical Parameters .....	26

RESULTS AND DISCUSSION .....	27
Results of Laboratory Studies .....	27
Abrasion and Leaching Results .....	27
Quantitative Removal of Rare-earth Label .....	28
Comparison of Labeled and Natural Sand .....	32
RESULTS OF FIELD EXPERIMENT .....	32
CONCLUSIONS .....	44
REFERENCES CITED .....	47
APPENDIX .....	50
Table 1 .....	50
Table 2 .....	51
Table 3 .....	52
Table 4 .....	53
Table 5 .....	56
List of Tables .....	v
List of Figures .....	vi

## LIST OF TABLES

Table No.	Description	Page
1	Sand abrasion results .....	50
2	Seawater leaching results .....	51
3	Acid stripping and fluoride precipitation results .....	52
4	Tabulated field data and corrected $A_0$ values .....	53
5	Physical and meteorological parameters day of tracer study .....	56

## LIST OF FIGURES

Figure No.	Description	Page
1a	Rotary evaporator and abrasion cylinder.....	15
1b	Coring device and sample rack .....	15
2	Flow chart showing sample preparation .....	16
3	Curve used to determine $A_0$ .....	19
4	Location map .....	22
5	Swell diagram, Virginia Beach, Va. ....	23
6	Beach profile, Camp Pendleton Beach .....	24
7	Control grid showing sample locations .....	25
8	Dry sieve analysis: labeled(lmg/gm) vs natural sand .....	29
9	Dry sieve analysis: labeled(lmg/gm) vs natural sand .....	30
10	RSA analysis: labeled(lmg/gm) vs natural sand .....	31
11	RSA analysis:labeled (lmg/gm) vs natural sand .....	33
12	RSA analysis: labeled(lmg/gm) vs natural sand .....	34
13	RSA analysis: labeled(10mg/gm) vs natural sand ....	35
14	RSA analysis: labeled(lmg/gm) vs labeled(10mg/gm) ..	36
15	Dry sieve analysis: Camp Pendleton sand before labeling, after labeling and natural sand day of tracer study .....	37
16	Tracer dispersal pattern 1 hr after injection .....	39
17	Tracer dispersal pattern 4 hr after injection .....	40
18	Weight percent labeled sand conversion graph .....	41
19	Mean $A_0$ values plotted against sample line .....	43



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## INTRODUCTION

### General Statement

The movement of sand in the nearshore zone has been a matter of great interest during the past few decades. Beach and nearshore sand movement, as a geological process, has prompted transport studies which have been concerned with such diverse phenomena as channel maintenance, flood protection, beach erosion and accretion and recreation. In general, knowledge of the processes responsible for the dispersion of sand along the bottom is somewhat incomplete. Interactions between waves and bottom materials have been studied very extensively in the laboratory using wave tanks. Scaling problems usually render the extrapolation of experimental results to natural phenomena invalid. Another method which is frequently employed in sediment transport studies involves the use of theoretical models constructed from the empirically derived relationships existing between water particle motion and entrained sediment. These models have often been used to predict beach and offshore processes when actual field studies were unavailable. The lack of field verification tends to render the theoretical model somewhat invalid as a predictor of sediment transport processes. Field investigations constitute the final approach to sediment transport studies. Numerous coastal sediment transport studies have been initiated within the past few years but these have been rather general and qualitative due to the lack of accurate and sensitive methods.

### Previous Studies

The earliest attempts to study sediment movement often involved the use of ground glass as a tracer. This method was abandoned when it was found that the shape, density and statistical size variations did not conform exactly with the properties of the natural components in the sand. Later attempts involved the utilization of some natural component in the sand itself, such as a type of heavy mineral, which could be treated to render it readily distinguishable from the untreated grains. This approach was also found to contain several inherent problems, the most important of which was the fact that such a method could only be applied qualitatively if the tracer particle concentration could not accurately be measured before and after the experiment.

Goldberg and Inman (1955) suggest that a natural tracer of sand movement should be (1) of the same size distribution, density and shape as one of the major components under study, (2) easily and rapidly distinguishable from the sand mass, (3) available in relatively large amounts, (4) inexpensive and (5) able to retain its distinguishing properties over times comparable to the times of the fluid processes.

### RADIOISOTOPE STUDIES

The application of radioisotopes and radioactive labeling to study sedimentary processes has received much attention during the past few years. Goldberg and Inman(1955) used phosphorous-32 which is produced in quartz subjected to thermal neutron irradiation. This isotope emits 100% beta particles and labeled grains were detected by using a photo-sensitive plate. The expected sensitivity of this method was one part

per million. Inman and Chamberlin(1959) however, later found that the detection sensitivity of this method was only one particle per 100,000 particles but that greater sensitivity could be achieved by increasing the sample size and the size of the photosensitive plate. Inose, et al. (1955) and Sato, et al.(1961) have used radioactive glass sand to study sediment movement along sections of the Japanese coast. Reid(1958) fused scandium oxide with boron free soda glass which subsequently was ground and sieved and irradiated with pile neutrons. The radioactive glass sand was then placed on the sea bed near the coast of Norfolk, England. Arlman, et al (1958) have studied sediment transport in the Netherlands by incorporating scandium-46 into an ion exchange resin. The zeolite chosen, Ionac C50, an inorganic resin has a density of 2.75 to 2.78 which closely approximates the sand along the coast of the Netherlands. It was found that the hardness of the artificial resin was far below that of natural sand but that heating to 900°C increased the hardness of the resin to a point where it could be used as a tracer material. Svasek and Engel (1961) have employed scandium-46 to study sediment transport at the entrance to the Rotterdam waterway. Taney (1962) has used ground radioactive glass in an attempt to qualitatively determine the path of beach sediments in a labotatory test basin. Bonefille, et al.(1966) have used radioactive gold-198 to evaluate sediment transport in the Gironde estuary. Courtois and Monaco (1969) have used natural sand coated with radioactive chromium-51 to evaluate coastal sediment drift rates along the coast of France north of Barcares. Duane and Judge (1969) describe the development of a versatile field survey system which could be used with a variety of radio-nuclides. In this study attempts were made to correlate the observed

sand transport phenomena with wave and current variables at the test site. Xenon-133 was the isotope employed and a comparison of fluorescent, radioisotopic, stable isotope and natural mineral tracing methods is presented as an appendix.

The disadvantages encountered when using radioisotopes include the possible damaging effect of radiation to the investigators and the extent to which the method could be applied in public areas without the creation of a health hazard, either real or imagined. Increased public concern within the past few months over pollution and contamination of our natural waters and beaches may preclude the application of radioactive materials in areas that are frequented by the general public.

#### FLUORESCENT STUDIES

Another method by which sand grains have recently been labeled for transport studies involves the utilization of a fluorescent dye. A thin coating of the dye is applied to a selected number of sand grains and then reintroduced to the natural environment. Since its introduction in 1958 by the Russians, this technique has received considerable attention. This attention stems primarily from the fact that no health hazard is encountered, the materials and equipment needed to label, detect and count the tracer grains are relatively simple and inexpensive. Ingle (1966) presents, as an appendix, several fluorescent formulations with costs ranging from U.S. \$0.12 to \$0.20/lb. The sensitivity of this method has been reported by Zenkovitch (1960) to be one part per billion, however most studies have reported sensitivities in the part per million range (Ingle 1966). Discrimination of the labeled grains is normally accomplished

by visual counting either under white or ultraviolet light. Teleki (1967) describes an automated fluorescent detection apparatus using an optical system coupled with a photomultiplier tube; however visual counting remains the most often used means of detection. Whatever the method of detection may be, it is subject to error from naturally occurring background fluorescence, especially carbonate materials.

A potentially more serious problem, as suggested by Malone (1969), is the alteration of the surface of the sand grains after they have been treated with an organic dye and bonding agent. This alteration may significantly affect the hydraulic properties of the tracer sand thereby making the interpretation of results difficult. Ingle (1966) presents data to indicate that no such alteration occurs over times comparable with those of the fluid medium, but he mentions that a liquid detergent must be applied to the fluorescent grains prior to their release at the test site. Apparently the organic coating is somewhat hydrophobic and the detergent acts to suppress the bouyant tendencies of the grains. If this is true, there exists a doubt as to whether the fluorescent grains effectively duplicate unlabeled grains in their natural enviornment. Duane and Judge (1966) indicate that fluorescent coatings on sand grains can undergo both chemical and physical degradation. The labeled grains then loose their identity when mixed with the natural sand. Additional studies involving the use of fluorescent tracers have been reported by Vernon (1966), Yasso (1966), Schwartz (1966), Teleki (1966) and Boon (1969).

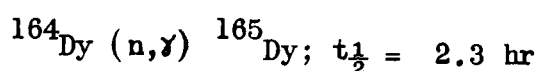
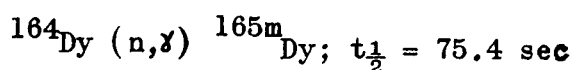
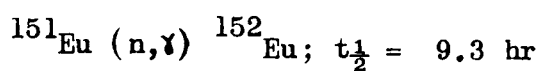
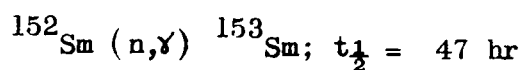
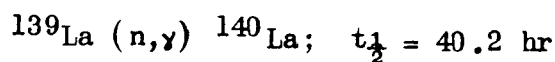
#### STABLE ISOTOPE METHODS

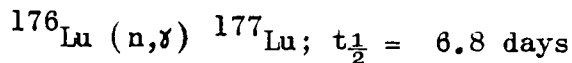
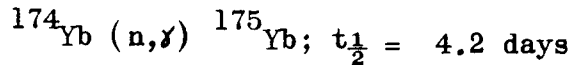
It can be seen from the above discussion that there is a need for an

accurate, sensitive and rapid means by which sand grains can be labeled and their movement on the beach and nearshore regions be analyzed. The method should be sensitive in the part per million range, thereby equalling or surpassing the sensitivities of the previously described techniques. Malone (1969) has reported a technique employing stable isotopes, as labels, which can upon recovery be determined by neutron activation analysis. This method involves (1) the identification of a tracer which is either absent or present at very low concentrations in the natural environment, (2) the development of a suitable means by which the tracer can be affixed to the sand grains without altering their size or properties and (3) the development of a method by which the tracer upon recovery, can be qualitatively and quantitatively measured.

The rare-earth elements (Atomic numbers 57-71), as suggested by Malone, can be employed as a tracer material since they have low concentrations in sediments and seawater (Haskin and Gehl (1962), Goldberg, et al. (1963), Coryell, et al. (1963), Schofield and Haskin (1964), Wildeman and Haskin (1965), Goodell (1967), and Høgdahl, et al. (1968)). In addition, the rare-earth elements have isotopes which can be measured in the part per billion range through neutron activation analysis.

Malone (1969) considered the following rare-earth isotopes:





In the above section, the stable parent nucleus is listed on the left of the equation, in parenthesis are listed the activating particle(neutrons) and the type of particles emitted(gammas). Following the parenthesis, the radioactive daughter and its half-life are listed.

Activation times varied from 30 seconds to 20 hours in order to produce a gamma spectrum in which the rare-earth isotope activity could instrumentally be discriminated from the untreated sand. Of the six stable isotopes reviewed, he selected europium because it could be analyzed immediately after activation. Post activation cooling has the tendency to reduce the rare-earth tracer activity and thereby reduce the analytical sensitivity.

Field tests were carried out by Malone on the beach at Galveston, Texas and the results indicated a detection limit of about 3 or 4 sand grains per 100,000. Presumably the reduction in sensitivity is explained by interfering activity produced within the untreated sand as a direct result of long irradiation times. He concludes by stating that the sensitivity of the stable isotope method may be extended by increasing the amount of rare-earth label per unit weight of natural sand. Increasing the amount of rare-earth label tenfold, from the 1mg/gm level to the 10mg/gm level, may give the desired detection limits but may also introduce other problems. The most important of such problems would be the alteration of the surface properties of the sand grains which would tend to affect the hydraulic behavior of the grains. Malone did not present any data which would indicate that the stable isotope label at



the  $\text{lmg/gm}$  level did not affect the hydraulic properties of the grains, nor did he show that the refractory oxide coating remained on the grains after abrasion on the sea bed or leaching in the seawater itself. He found that a 10 minute activation of one gm of labeled sand did not produce sufficient rare-earth activity to be discriminated instrumentally from the untreated sand. In addition, to eliminate the excess activity produced by the sand itself, a pre-activation concentration procedure was adopted in which the rare-earth label was acid stripped from the grains and precipitated as a fluoride before counting. No data was presented which would indicate that this analytical procedure provided for quantitative removal and recovery of the tracer label, an essential requirement if this method is to be valid.

#### Research Approach

In view of the need for rapid, accurate and sensitive means of labeling sedimentary particles, rare-earth stable isotopes would appear to warrant further studies. Dysprosium-164 is a rare-earth element which might be used to improve upon the stable isotope tagging method. Dysprosium-164 has a metastable activated product, Dy-165m, with a half-life of only 75 seconds and can be detected in the part per billion range after a 10 second irradiation with a neutron flux of  $10^{13}$  neutrons/cm<sup>2</sup>/sec. A short activation time such as 10 seconds has the tendency to suppress the buildup of activity of interfering isotopes in the sample and consequently allow the sample to be counted immediately after activation. Tests will be conducted to determine whether or not the isotopes of dysprosium produced in a thermal neutron flux (mean energy of 0.025eV)

will be detectable over any other isotopes produced within the sand sample.

A sophisticated data collection and presentation system could be developed once the above criteria are met. This system would allow rapid counting of one gram portions of sand samples taken from a study area. The data obtained on a multichannel analyzer could be stored on magnetic tape or punched cards which can in turn be coupled to a computer, programmed to detect photopeaks of known energies, eliminate background radiation and measure counts within a given channel. Automatic data processing could even be extended to the point where an x-y plotter would be employed to construct observed dispersal patterns of the tracer material under a given set of wave and current parameters. Ideally, a sample from the field could be washed, dried, weighed and placed in a polyvial. The sample could then be transferred to a reactor which is equipped with a pneumatic transfer system, activated and counted with a multichannel analyzer whose output can be placed on magnetic tape to be later reduced. The increased efficiency and accuracy of data handling with automated systems, coupled with the increased sensitivity of the stable isotope method make it competitive with other previously described methods. In light of the potential promise of the stable isotope methods, further studies should be conducted to evaluate the applicability of these labeling procedures to sediment transport problems.

It is the twofold purpose of this study to appraise the use of dysprosium as a stable isotope which might be used in sediment transport studies and to investigate the physical and chemical properties of rare-earth oxide coatings on sand grains.

## LIMITATIONS

The use of stable isotopes for tracing the movement of sedimentary particles in the natural environment may have some limitations. Clay sized particles, whose diameters are less than 62.5 microns, cannot suitably be labeled with a stable isotope such as dysprosium. The excessive heat required ( $800^{\circ}\text{C}$ ) to form the refractory rare-earth oxides can cause clay minerals to lose pore water, adsorbed water and eventually, hydroxyl structure water. (Grimm, 1962 p. 38) This apparent collapse in structure would preclude the application of stable isotopes as a possible label for this group of sedimentary particles.

Those beach and nearshore beach deposits rich in monazite, xenotime and other heavy minerals such as bastnaesite and euxenite could, only under certain conditions, be selected as possible study areas due to the potential background interference caused by the rare-earth phosphates found in this particular heavy mineral suite. Certain granitic intrusives and granitic gneisses that occur in two belts of the Piedmont province in Virginia, North Carolina, South Carolina, Georgia and Alabama are the principal bed-rock sources of monazite in the southeastern states. (Mertie, 1953). Those streams originating in the monazite bedrock are likely to contain large amounts of monazite in their headwaters and possibly for considerable distances downstream. In South Carolina, Georgia and Florida, there exist many streams which originate in the monazite belts of the Piedmont. These streams have transported a large volume of heavy minerals to the Coastal Plain during Pleistocene interglacial stages. Beach deposits in these areas may range from 0.01 to 0.1 percent heavy minerals, while monazite constitutes approximately 5% of the heavies. (Mertie, 1953). Monazite

is typically about 0.30%  $Dy_2O_3$ . (Woyski and Silvernail, 1961 p.519)  
Economically these concentrations are of little significance, however, they are critical in judging the suitability of a study area for stable isotope tagging.

An additional limitation inherent in this method is the cost of tracer preparation and sample analysis when compared with fluorescent methods of tracer preparation. Stable isotope tracer preparation costs approximately \$4.50 per pound (10 mg label per 1 gm sand). Justification of the additional costs associated with the stable isotope method would be contingent upon the detection limit achieved.

## METHODS

### Laboratory Methods

#### SAND PREPARATION

Approximately 30 lbs of sand were removed from the swash zone at Camp Pendleton Beach. The sand was washed, wet sieved and placed in a pan of 1N HCl (J.T. Baker Chemical Co.) and allowed to stand overnight to remove the carbonate materials. After rinsing, the sand was placed in a solution of Chlorox and allowed to stand overnight to remove any organic coatings on the sand grains. The sand was then re-washed with distilled water, placed in a Calgon solution and allowed to stand for two hours to flocculate the clay and silt sized particles. The sand was then washed three times with tap water, twice with distilled water, dried and allowed to pass through a 2 mm sieve to remove any larger sized particles.

#### LABELING PROCEDURES

Ten gm of  $\text{Dy}_2\text{O}_3$  (Spex Industries, Inc.), with a spectrographic purity rating of 99.9% was dissolved in 25 ml of concentrated  $\text{HNO}_3$  (J.T. Baker Chemical Co.) and then diluted to 260 ml. One kilogram of prepared sand was placed in a 2 qt pyrex dish and mixed with 260 ml of  $\text{Dy}(\text{NO}_3)_2$  solution, placed in an oven at  $120^\circ\text{C}$  and dried. Drying was terminated when all visible traces of liquid were gone. The dried sand was then placed in Coors porcelain evaporating dishes and in a muffle furnace at  $800^\circ\text{C}$  for 45 minutes to convert the soluble nitrate to a water insoluble refractory oxide coating on the sand.

## EVALUATION OF EFFECTIVE DIAMETER AND SETTLING PROPERTIES OF LABELED SAND

The degree to which the grain size frequency distribution of labeled sand grains duplicated the natural sand was determined by sieving and by settling methods. Quarter phi screens ranging from -1.0 to 4.0 were employed in the sieve analysis of both labeled and natural sand to determine the extent to which, if any, the effective diameters of the grains were altered by the application of the rare-earth label. The rapid sediment analyzer used (Benthos Inc., Model 3410) consisted of a water filled vertical tube, approximately one meter long, into which a 2 gm sand sample is introduced. As a sediment sample enters the tube, the effective density of the water column increases abruptly, causing an increase in the pressure read by a transducer mounted near the base of the tube. A signal from the transducer is sent to an X-Y plotter which generates a settling curve that is a function of the samples grain size distribution. Further discussion of RSA principles and application can be found in Schlee (1966) and Sanford (1970).

## ABRASION AND LEACHING STUDIES

An isotope of dysprosium with a suitable half-life was not available for laboratory evaluation. Instead, radioisotopes of lanthanum and samarium (La-140,  $t_{1/2} = 40.2$  hr and Sm-153,  $t_{1/2} = 46.7$  hr) which were produced at the Reactor Facilities of the University of Virginia and Virginia Polytechnic Institute, respectively, were employed to label sand to determine tracer loss through abrasion and seawater leaching. These substitutions were made on the assumption that the rare-earth elements are chemically similar.

Thirty mg of radioactive lanthanum were applied to 30 gm of sand using the previously described methods. This provided a coating ratio of 1 mg La/gm sand. In order to evaluate a higher coating level, one mg of radioactive samarium was mixed with enough stable samarium to coat 11 gm of sand or a ratio of 10 mg samarium per gm of sand. Aliquots of each type of labeled sand were separately placed in a 500 ml polypropylene bottle connected to a rotary evaporator (Fig. 1a). Fifty ml of seawater were added to the cylinder and the sand allowed to abrade for a period up to 46 hours. After the abrasion test the activity of the sand was compared with the activity of the seawater evaporate to determine the loss of tracer activity. Gamma spectra of the radioisotopes were instrumentally measured on a multichannel analyzer (Victoreen, Model PIP 400). Sand abrasion results are listed in Table 1.

Seawater leaching tests were also conducted by taking 1 gm portions of both lanthanum and samarium labeled sand and placing them in 100 ml beakers with 25 ml of seawater. After 46 hours, the sand was separated from the seawater by filtration and the activity of the sand was compared with that of the seawater evaporate to measure the loss of tracer through leaching. Gamma spectra again were measured on a multichannel analyzer. Seawater leaching results are found in Table 2.

#### PREPARATION OF SAMPLES FOR ACTIVATION

The field samples were processed according to the sequence shown in Figure 2. Approximately 2.0 gm of sand was extruded from each core sample into a small beaker. The sand was washed three times with tap water and once with distilled water. Ten mls of 1N HCl were added to each beaker and the solution was allowed to stand overnight. The sample was then

Fig. 1a (top) Picture showing rotary evaporator and  
abrasion cylinder used for abrasion  
studies

Fig. 1b (bottom) Picture showing sample rack holding  
plastic coring devices



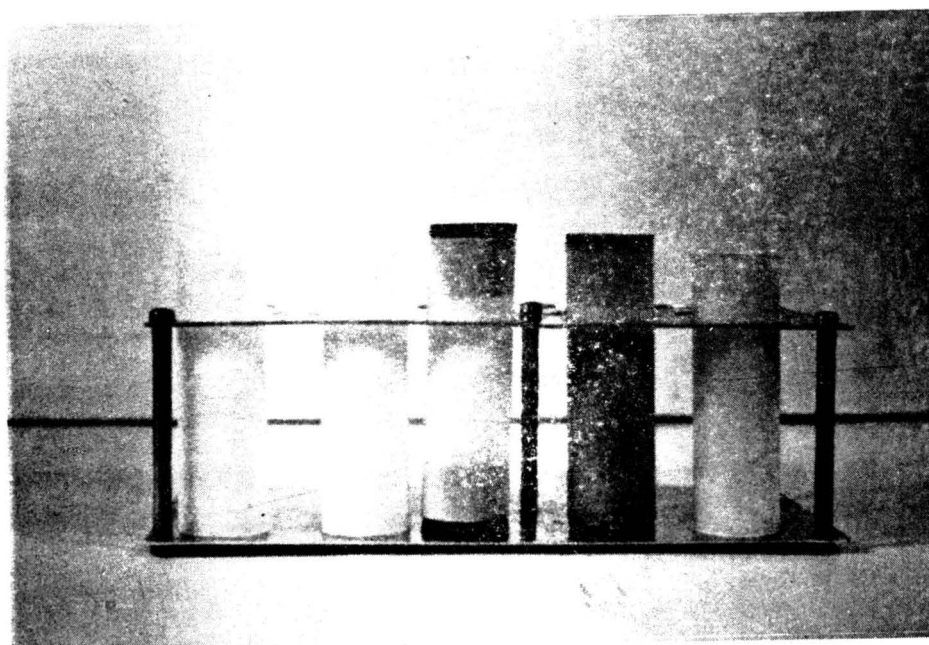
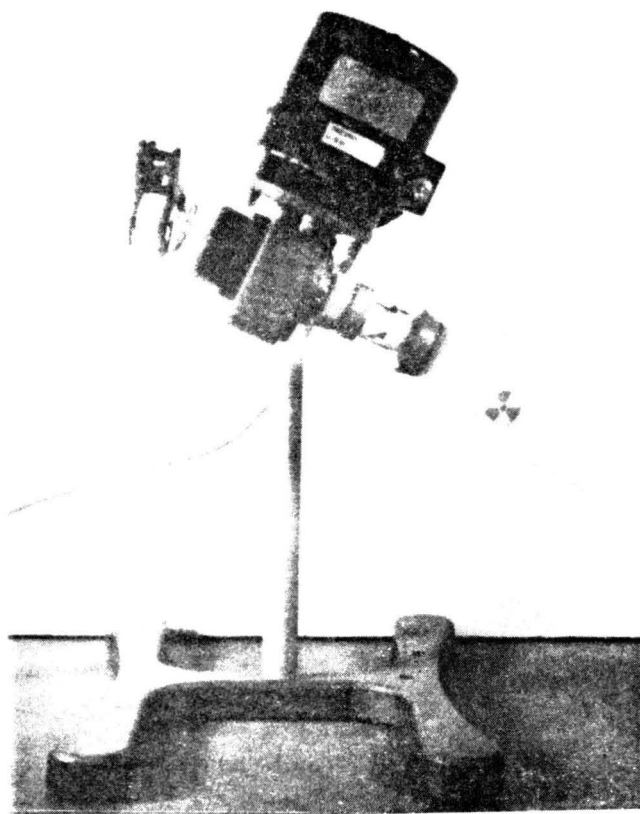
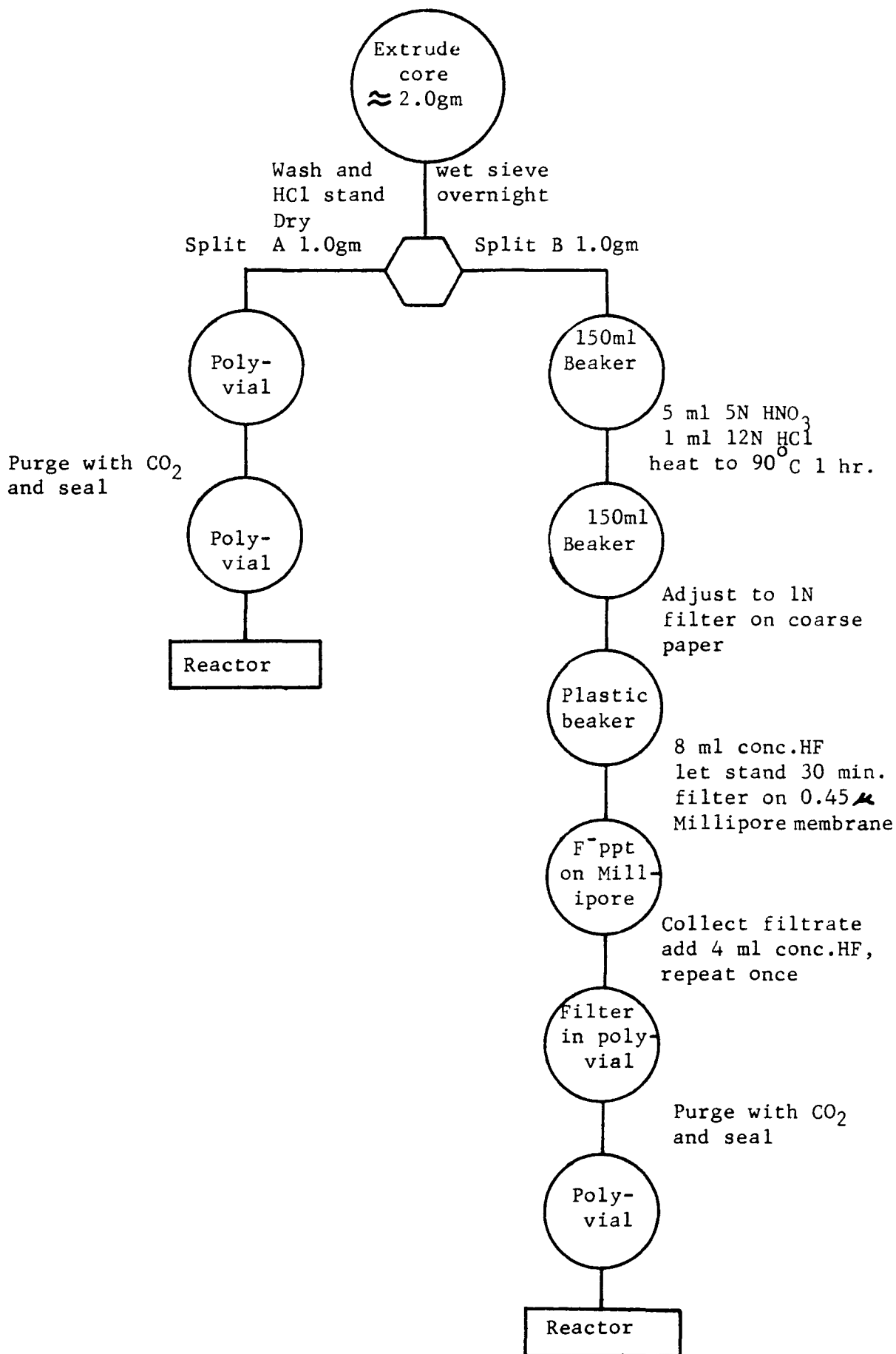


Fig. 2 Flow chart showing two methods by which  
samples were prepared for neutron activation



washed several times, dried, split and weighed on a Mettler balance. One gm was placed in a polyvial, purged with  $\text{CO}_2$  and sealed for activation. An additional one gm aliquot was placed in a 150 ml beaker to which 5 ml of 5N  $\text{HNO}_3$  (J.T. Baker Chemical Co.) and 1 ml of 12N  $\text{HCl}$  (J.T. Baker Chemical Co.) were added. The solution was maintained at  $90^\circ\text{C}$  for 1 hour. The normality of the acid solution was adjusted to 1N and filtered on Whatman #4 filter paper to remove the sand grains. The filtrate was retained in a plastic beaker. Eight mls of 49%  $\text{HF}$  (J.T. Baker Chemical Co.) were added and the solution was allowed to stand for 30 minutes. The solution was then filtered on 0.45  $\mu$  Millipore filter membrane. Four mls of 49%  $\text{HF}$  were added to the filtrate and the solution was refiltered through the same filter membrane. Four additional mls of 49%  $\text{HF}$  were added and the filtrate was refiltered a third time. The Millipore filter containing the rare-earth fluoride precipitate was placed in a polyvial, purged with  $\text{CO}_2$ , and sealed for activation. Approximately 20 hours were required to prepare 100 samples for activation, using the above procedures.

#### EVALUATION OF STRIPPING PROCEDURES

In order to evaluate the effectiveness of the above mentioned stripping procedures, five 1 gm aliquots of Sm-153 labeled sand and three 1 gm aliquots of La-140 sand were used to determine the extent to which the rare-earth coating could be removed from the sand. Both samarium and lanthanum tracer sand were labeled in the ratio of 10 mg rare-earth per gm sand. Quantitative removal of the rare-earth label and its recovery in a form suitable for activation was a necessary prerequisite if this procedure had to be followed in the analysis of sand samples taken from the field. Acid stripping and fluoride precipitation results are listed in Table 3.

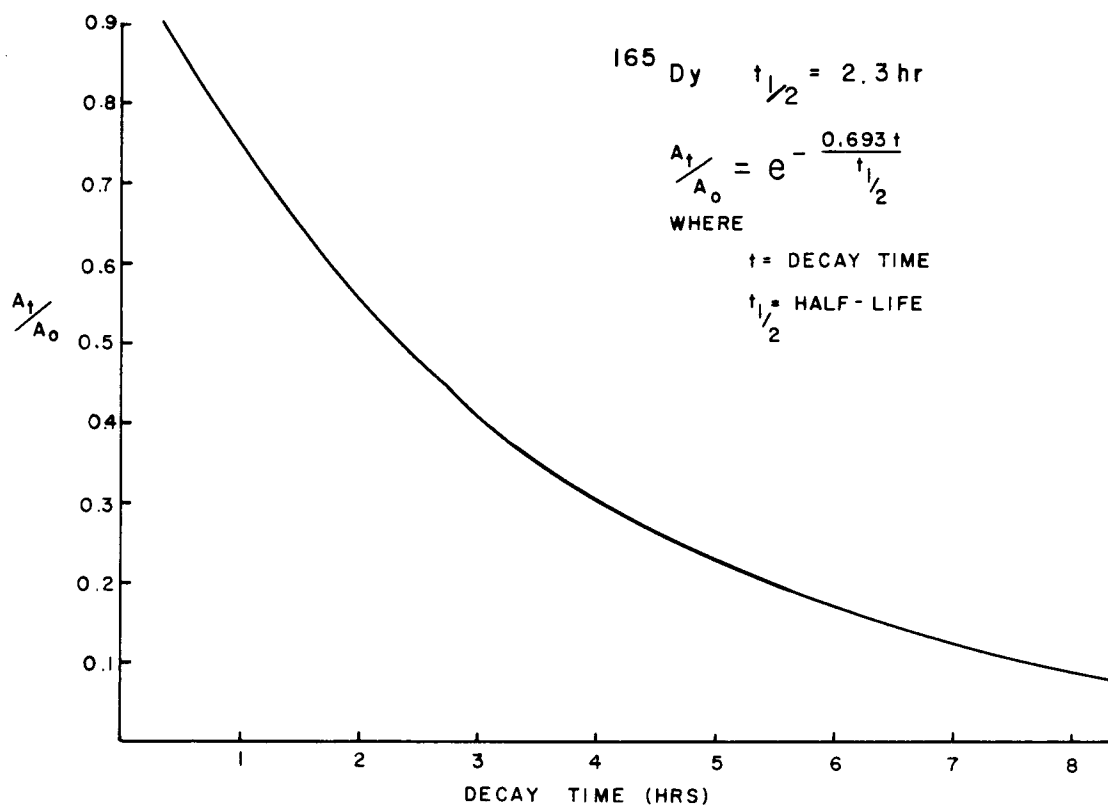
## ACTIVATION ANALYSIS OF FIELD SAMPLES

Field samples, after the above preparation, were irradiated at the Reactor Facility at Virginia Polytechnic Institute in Blacksburg, Virginia. This reactor produces a maximum thermal neutron flux of  $1.3 \times 10^{12}$  n/cm<sup>2</sup>-sec. Two irradiation facilities at the reactor were employed in this activation analysis.

The first facility was the pneumatic transfer system in which the sample to be activated is sent into the core of the reactor, activated for the desired length of time and then returned to the laboratory. Combined transit time to and from the reactor core using nitrogen gas as the carrier is approximately 5 seconds. This type of activation is particularly useful when analyzing short-lived isotopes such as <sup>165m</sup>Dy, which has a half life of 75 seconds. Isotopes with large cross sectional areas, such as dysprosium-164, are very susceptible to neutron irradiations of only a few seconds. In the present instance, sand samples were irradiated for two seconds and were counted immediately afterwards.

The second irradiation method employed at the reactor facility was bulk sample activation in one of several thermal neutron beam ports. Up to 150 samples were simultaneously irradiated for 2 minutes. The thermal neutron flux ( $\phi$ ) in the beam port was  $6.7 \times 10^{11}$  n/cm<sup>2</sup>-sec. After irradiation, these samples were allowed to cool for at least 2 hours before counting was initiated to eliminate the effect of interfering isotopes in the sample. A two hour delay before counting precludes the analysis of short-lived products such as the metastable form of dysprosium-165. Instead, the longer half life (2.3hr) <sup>165</sup>Dy is analyzed.

Fig. 3 Decay curve of 2.3 hr Dy-165 used to determine  $A_0$  values



Gamma radiation emitted by the activated samples was measured with a lithium drifted, germanium detector. Ionizing particles, such as neutrons, entering the semiconductor create many ion pairs. In germanium, about 3 eV is expended per ion pair so that a 3 meV event would give rise to about a million ion pairs. The collected ions represent a current pulse which can be amplified and, since the number of ions created is directly related to the energy of the ionizing event, the amplitude of the current pulses are related to the energy also. The amplified current pulses are sent to a multichannel analyzer whose function is to convert the analogue voltage signals representing discrete nuclear events into digital form. An Intertechnique, Didac 4000, multichannel analyzer was employed to count the activated samples. Only that portion of the analyzer memory between 0.095 meV and 0.108 meV was used to register counts. The Ge (Li) detector efficiency for this energy range was 7%. The samples were counted for two minutes after which the data was transferred to IBM cards. Samples were allowed to decay between 2 and 8 hours before counting. Approximately 5 hours were required to count 100 samples. All activities were standardized by using the  $A_t/A_0$  ratio obtained from Figure 3, in which activity at time out of reactor ( $A_0$ ) is given in counts per minute and  $A_t$  is the activity of the sample after decay time ( $t$ ).



## Field Methods and Procedures

### TEST SITE

Camp Pendleton Beach (Fig. 4) served as the site for a tracer study carried out on April 13, 1971. The study site lies immediately south of the Virginia Beach City property line and is situated on the Camp Pendleton State Military Reservation.

Tides in the vicinity of the study area are predominantly semidiurnal with a mean range of 3.4 feet and a spring range of 4.1 feet. Waves reach the beach predominantly from the northeastern sector in the winter and from the southeastern sector during the summer months. Swells reach Virginia Beach (Fig. 5) predominantly from the northeast and easterly directions with a range between 1 and 6 feet. Calms prevail approximately 19% of the time. (Saville, 1954 and Neumann and James, 1955)

The average foreshore slope at Pendleton Beach is  $6^{\circ}$  (Fig. 6) with sand size predominantly between 0.25 and 1.0 mm in diameter.

### SAMPLE GRID

The sample grid (Fig. 7), consisting of nylon lines, was placed on the beach so that A-K was parallel to the shoreline beginning at the approximate low tide line. Sample points 1-7 extended from the low water line out past the breaker zone at low water. The grid lines were marked at 25 foot intervals to indicate the points at which samples were to be recovered.

Fig. 4 Location map showing approximate point of  
tracer injection

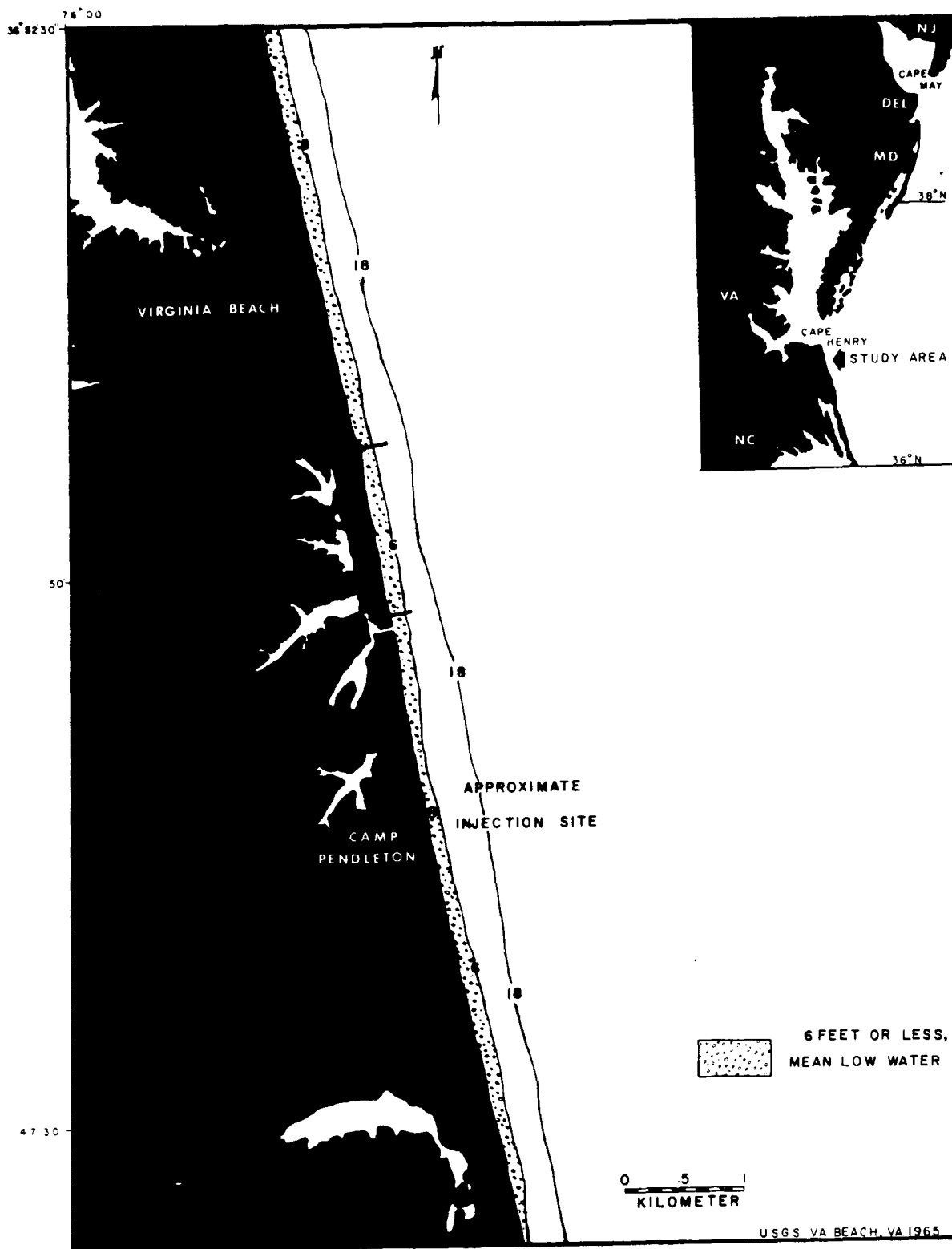
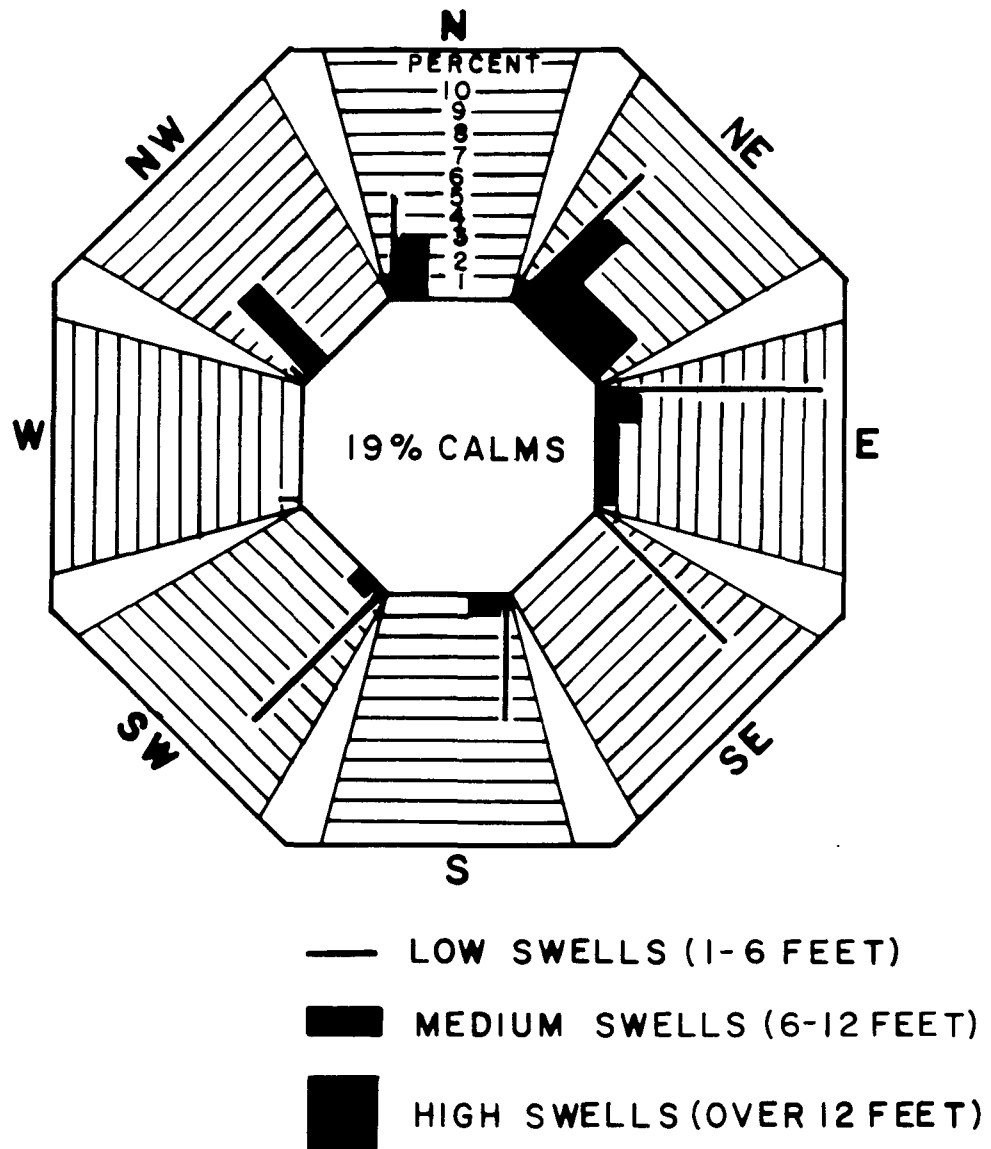


Fig. 5 Swell diagram, Virginia Beach, Va. ( U.S. Army  
Corps of Engineers)



**SWELL      DIAGRAM**  
**VIRGINIA BEACH, VIRGINIA**

Fig. 6 Beach profile Camp Pendleton Beach, Virginia  
April 13, 1971

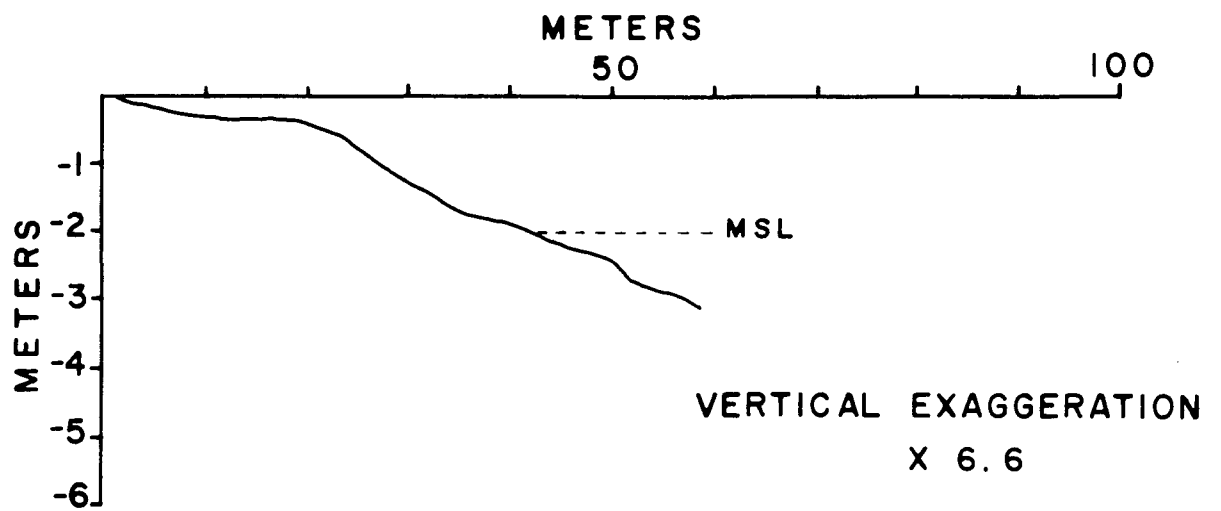
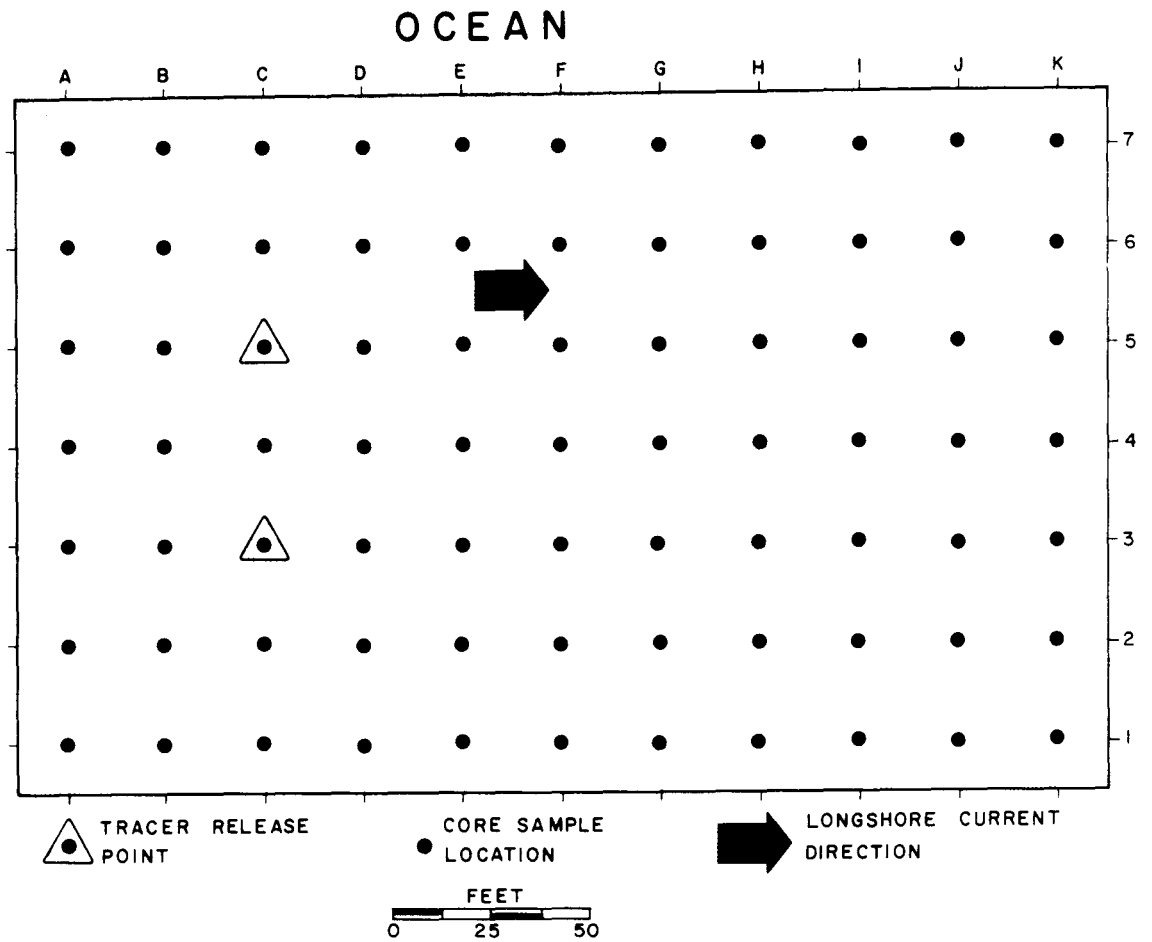


Fig. 7 Control grid showing points of tracer release,  
sample locations and direction of longshore  
current





## INJECTION OF TRACER AND RECOVERY OF SAMPLES

A total of 8.51 kg of tracer sand was placed on the bottom, 4.73 kg at point C-3 and 3.78 kg at point C-5. The sand was poured from plastic bags and evenly spread over a small area on the sea bed. One hour after tracer injection, the first set of samples were taken. Plastic tubes 10.16 cm long and 3 cm in diameter (Fig. 1b) were used to recover core samples over the sample grid. At the prescribed location, the tube was pushed into the bottom, removed and capped with rubber stoppers at each end. Two teams of two SCUBA divers each started sampling at opposite ends of the control grid. One diver would take a sample and then exchange it for an empty liner while proceeding to the next sample point. Each team proceeded seaward along one line until it had been completely sampled. The teams would return to the beach taking samples along the adjacent line. Upon returning to the beach, an empty set of core lines were obtained and the above procedure was repeated until the entire grid had been sampled. Four hours after tracer release a second set of samples was taken. The average time required to take one complete set of 77 samples was 1 hour.

## MEASUREMENT OF PHYSICAL PARAMETERS

Prior to tracer release the longshore current direction and velocity was measured on the surface by recording the time required for a grapefruit to travel 100 ft. During the experiment, the longshore current was measured with an electronic current meter (Bendix Marine Advisers, Model S240). Wave height, period and directions were visually recorded during the experiment at hourly intervals. Wind direction and velocity measurements were obtained from the U.S. Weather Bureau.

## RESULTS AND DISCUSSION

### Results of Laboratory Studies

#### ABRASION AND LEACHING RESULTS

Results of abrasion tests using radioisotopes of lanthanum and samarium are found in Table 1. It appears that the refractory oxide coatings on the sand grains are stable and resistant to abrasion at both coating levels studied. The experimental results show that after 26 hours of abrasion 4.1% of the lanthanum tracer activity had been lost. After 46 hours, 4.6% of the samarium tracer activity was lost through abrasion. These results suggest that the amount of tracer loss through abrasion is relatively small, at least for times up to 46 hours.

Results of the seawater leaching tests are found in Table 2. Less than one percent of the La-140 tracer was lost after 26 hours of seawater leaching in each of three samples. After 46 hours, approximately 5.8% of the total Sm-153 tracer activity was lost in each of three samples through similar tests. The combined effects of abrasion and seawater leaching were found to account for a maximum tracer loss of about 5%. Samarium oxide is known to be slightly more soluble than the oxide of lanthanum. This may account for the differential loss of samarium tracer through seawater leaching. The density of samarium oxide (7.43gm/cc) and lanthanum oxide (6.51g/cc) compares favorably with the density of dysprosium oxide (7.81g/cc). The three rare-earth elements have comparable electron configurations and are thought to have similar chemical properties. Based on these considerations, the substitution of La and Sm for Dy in the laboratory studies appears justifiable. AEC restrictions on the isotope

license available prohibited the production of large quantities of radioisotopes of lanthanum and samarium. Consequently, a limited number of laboratory analyses were performed with these isotopes. The results obtained do indicate that the refractory oxide label applied to the sand is relatively resistant to sea water leaching and abrasion, at least over time intervals comparable to those of the field test on the beach.

#### QUANTITATIVE REMOVAL OF RARE-EARTH LABEL

Acid stripping and fluoride precipitation results are found in Table 3. This stripping procedure was developed in the event that background activity was found to interfere with analysis. In this procedure the rare-earth label was stripped from the sand and then concentrated as a fluoride precipitate. During the early stages of the experiment several rare-earth concentration procedures were evaluated. These included hydroxide precipitation, oxalate precipitation, fluoride precipitation and an organic tri-butyl phosphate separation. In preliminary tests, the only procedure which provided quantitative recovery of the rare-earths was the fluoride precipitation. Radiosotopes of lanthanum and samarium were employed to determine the effectiveness of the acid stripping and fluoride recovery procedures. At least 90% recovery of the rare-earth fluoride was accomplished with both lanthanum and samarium isotope samples. Recovery of the rare-earth fluoride on the millipore filter membrane proved to be a suitable and convenient form for subsequent activation analysis.

Fig. 8 Dry sieve analysis comparison of natural and  
labeled (1mg/gm) sand

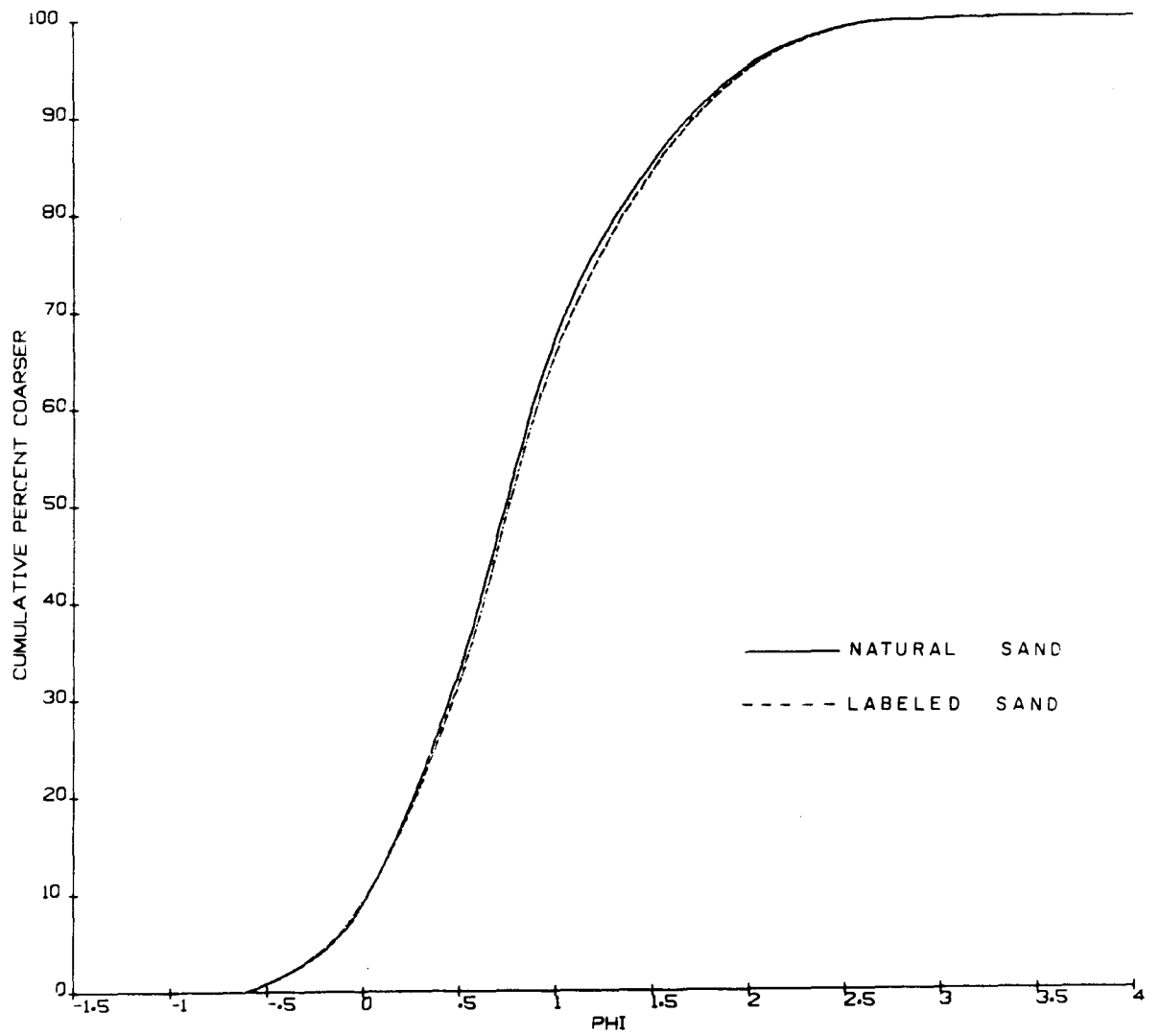


Fig. 9 Dry sieve analysis comparison of natural and  
labeled (1mg/gm) sand

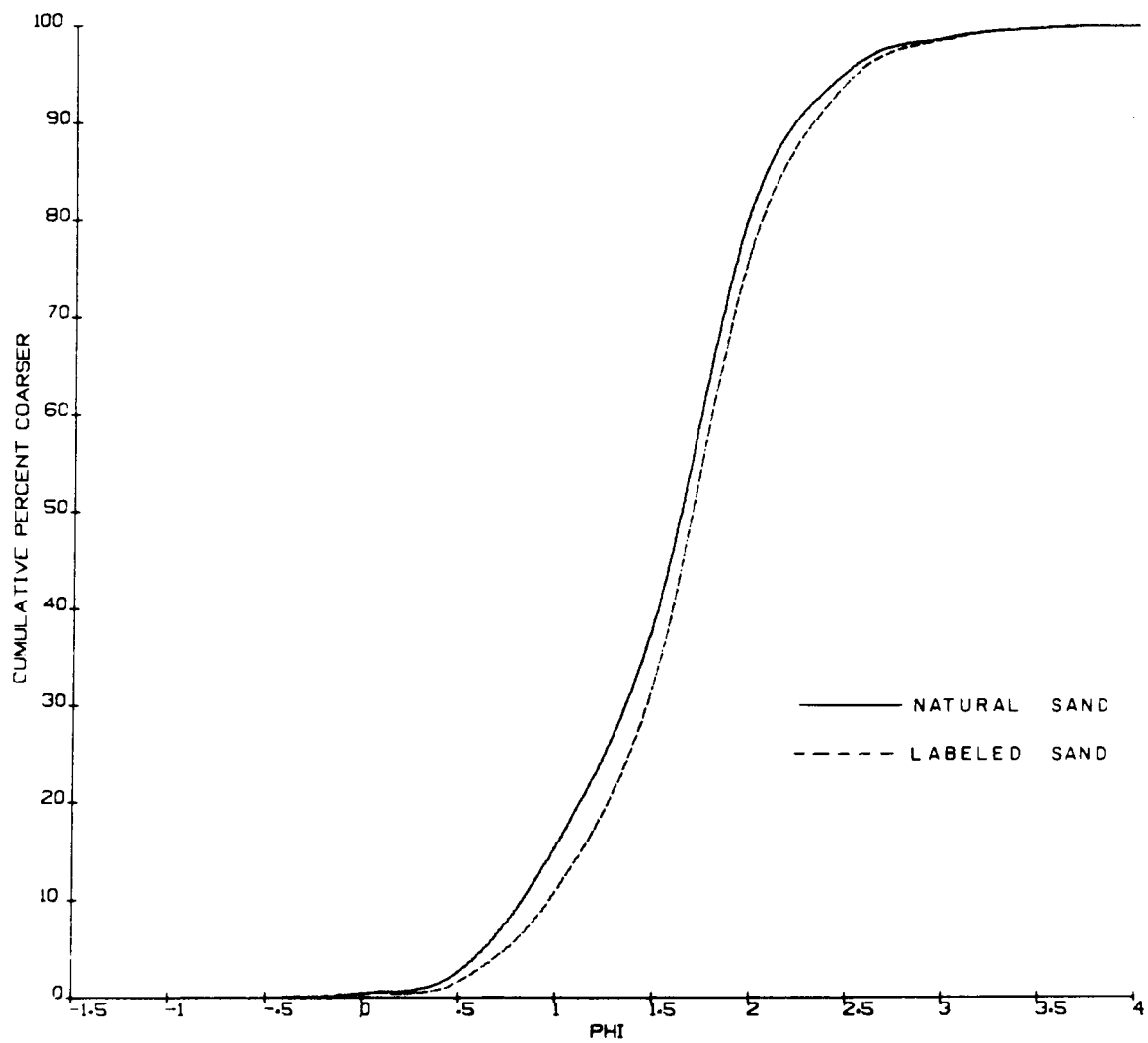
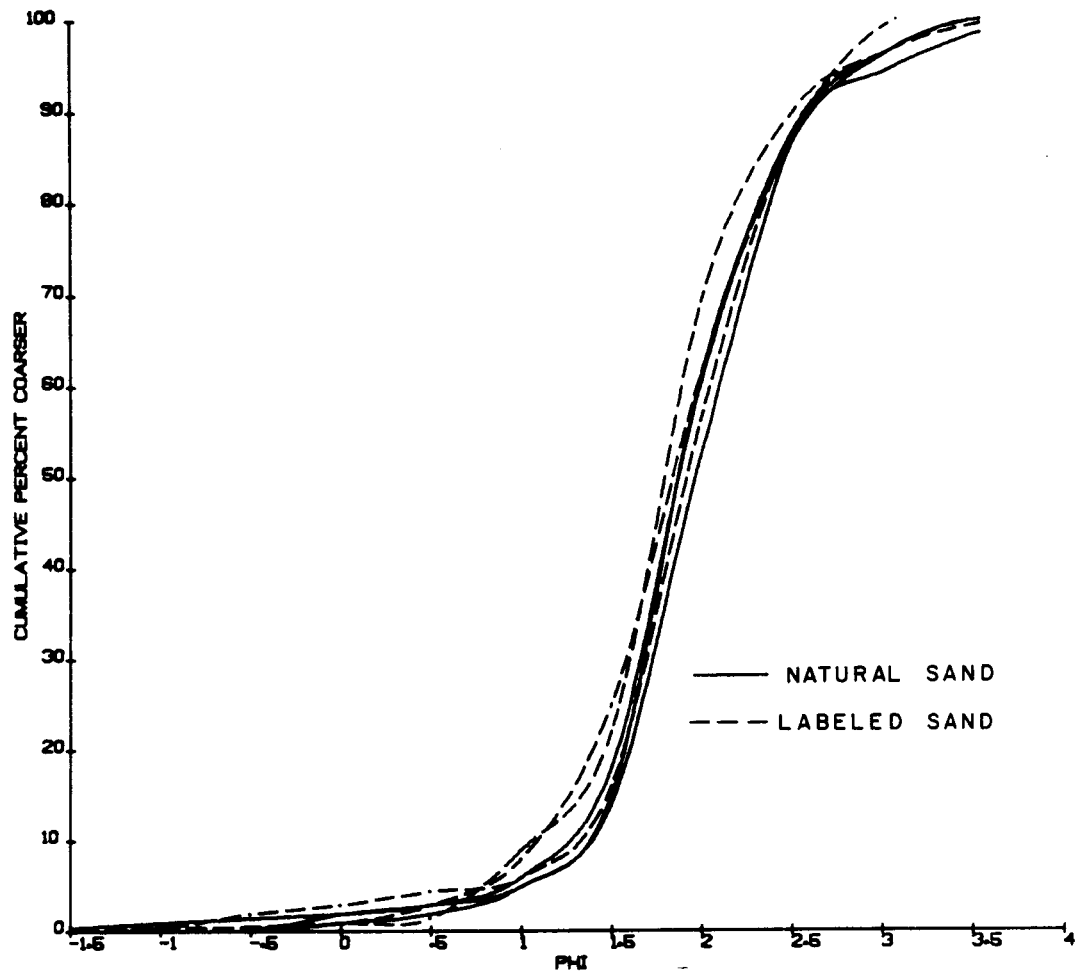




Fig. 10 RSA analysis comparison of natural and  
labeled (1mg/gm) sand



## COMPARISON OF LABELED AND NATURAL SAND

The settling properties and sieve diameter of the labeled tracer sand, applied at the 1 mg/gm and 10 mg/gm levels, were evaluated using both RSA and dry sieving techniques. A comparison between natural sand and the same sand labeled can be found in Figs. 8, 9, 10, 11, 12, 13, and 14. Figure 15 compares natural sand, labeled sand and Camp Pendleton sand on the day of the tracer study. The degree to which surface labeling alters such parameters as density, shape, size and roughness was qualitatively evaluated for both label concentrations (1 mg/gm and 10 mg/gm). The 10 mg level did not appear to significantly change any of the above mentioned properties when compared with either unlabeled sand or sand labeled at the 1 mg level. Based on this finding, the heavier tracer level was chosen for the field study. It was felt that by using this heavier coating detection, after dilution in the field, would be enhanced significantly.

### Results of Field Experiment

Tabulated field data are presented in Table 4. The  $A_0$  values for both sample series have been plotted on the control grid and are contoured (Figs. 16 and 17). These figures correspond to the first set of samples taken 1 hour after emplacement of tracer sand and the second set of samples taken 4 hours after emplacement. Thus the contour plots represent the dispersal patterns obtained at 1 and 4 hours respectively. Samples from Split B, stripped sand, were used to generate the above data points. The other fraction, Split A, was found to contain an excess amount of background activity, thereby making the interpretation of dispersal patterns

Fig. 11 RSA analysis comparison of natural and  
labeled (1mg/gm) sand

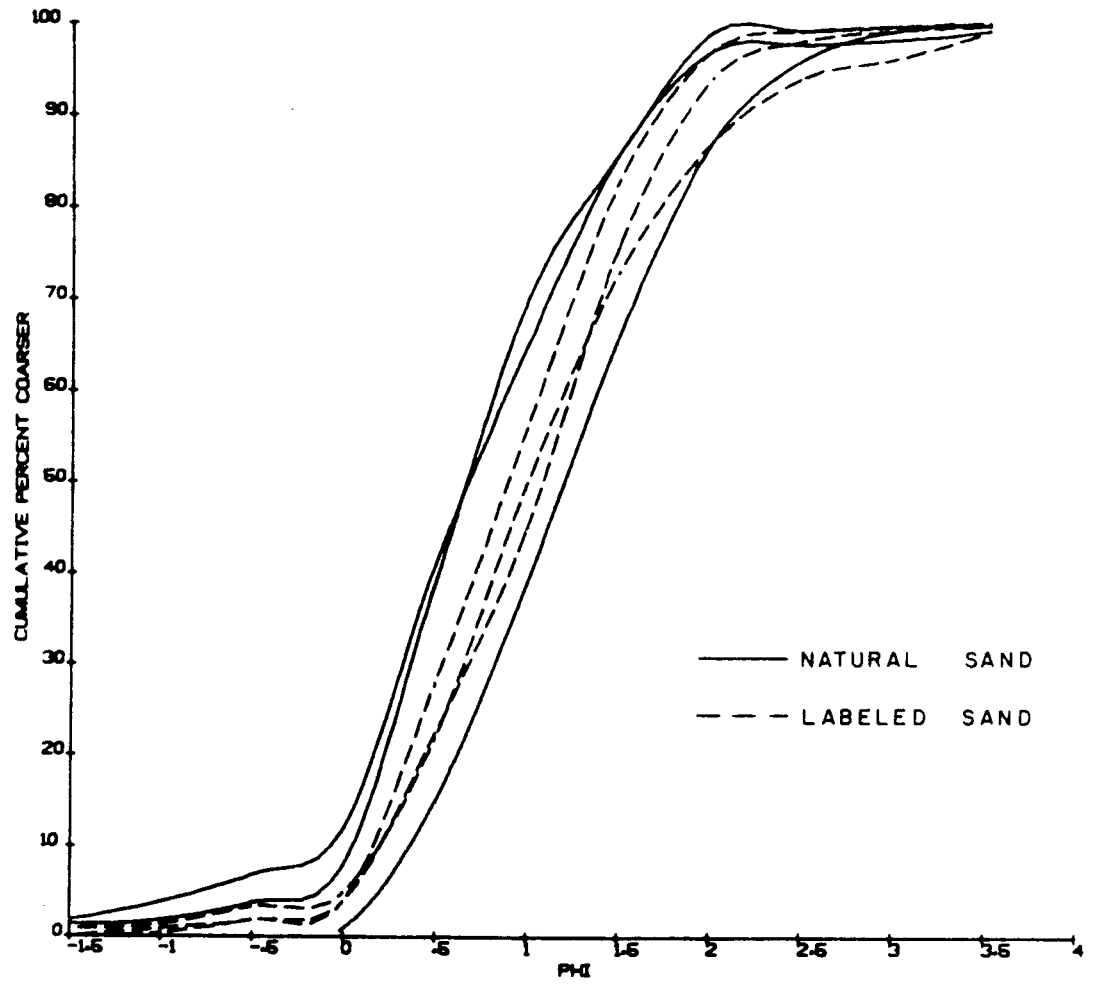


Fig. 12 RSA analysis comparison of natural and  
labeled (lmg/gm) sand

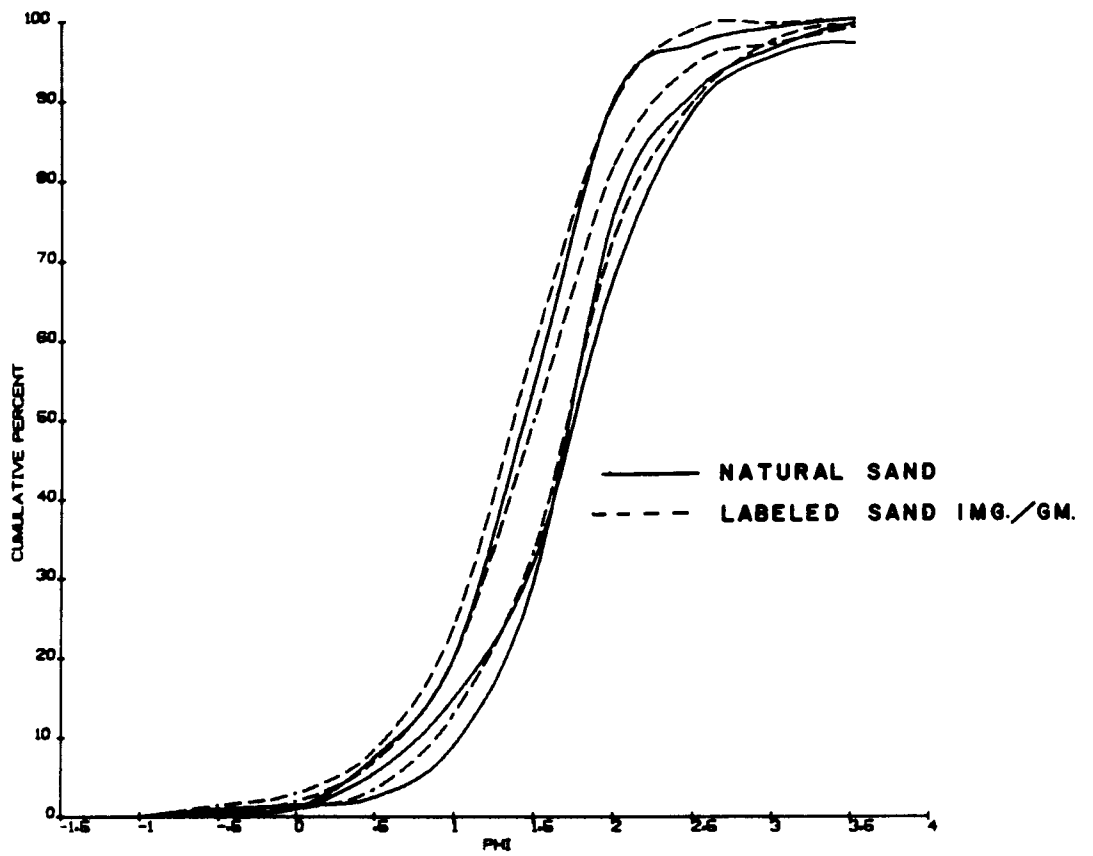


Fig. 13 RSA analysis comparison of natural and  
labeled (10mg/gm) sand



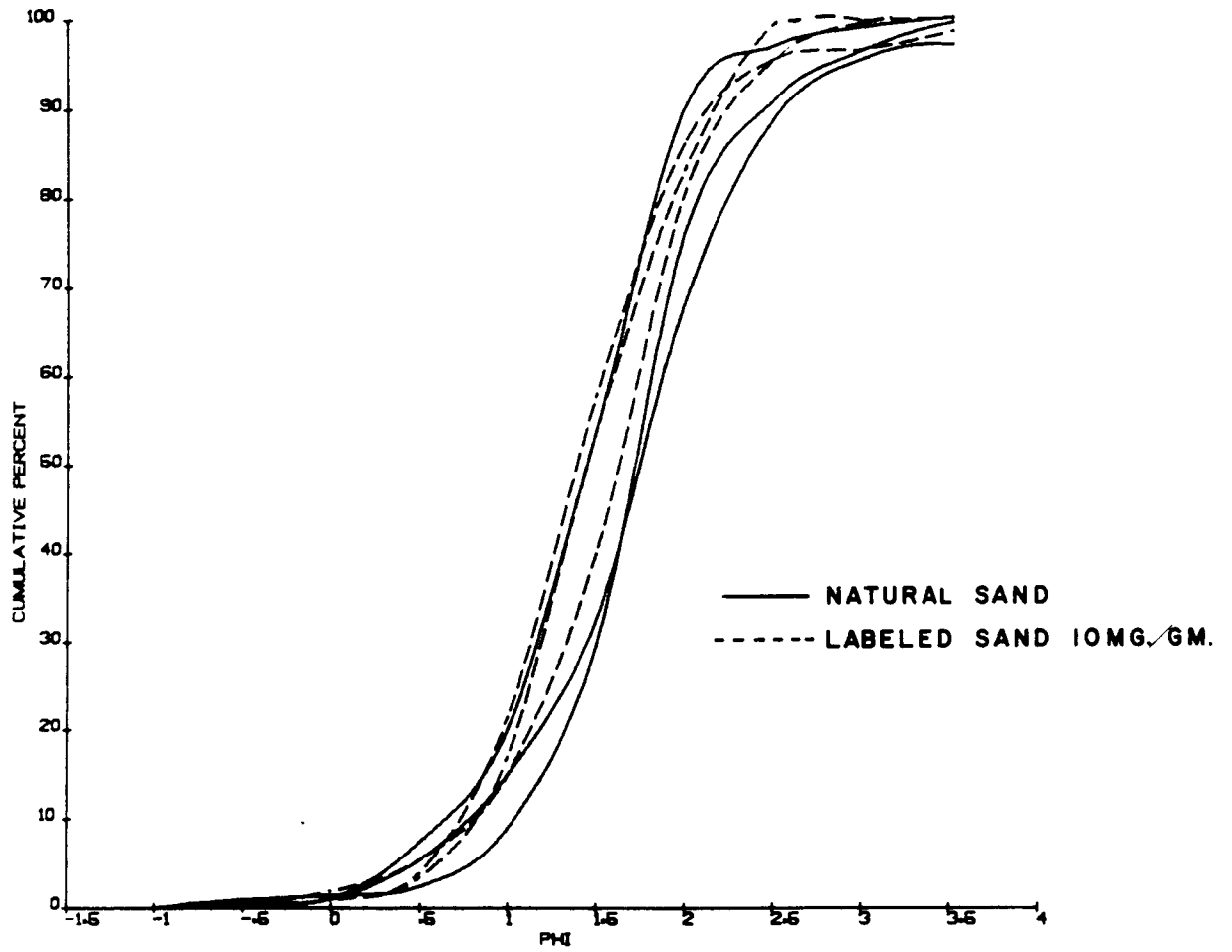


Fig. 14 RSA analysis comparison of sand labeled at  
both the 1 and 10 mg ratios

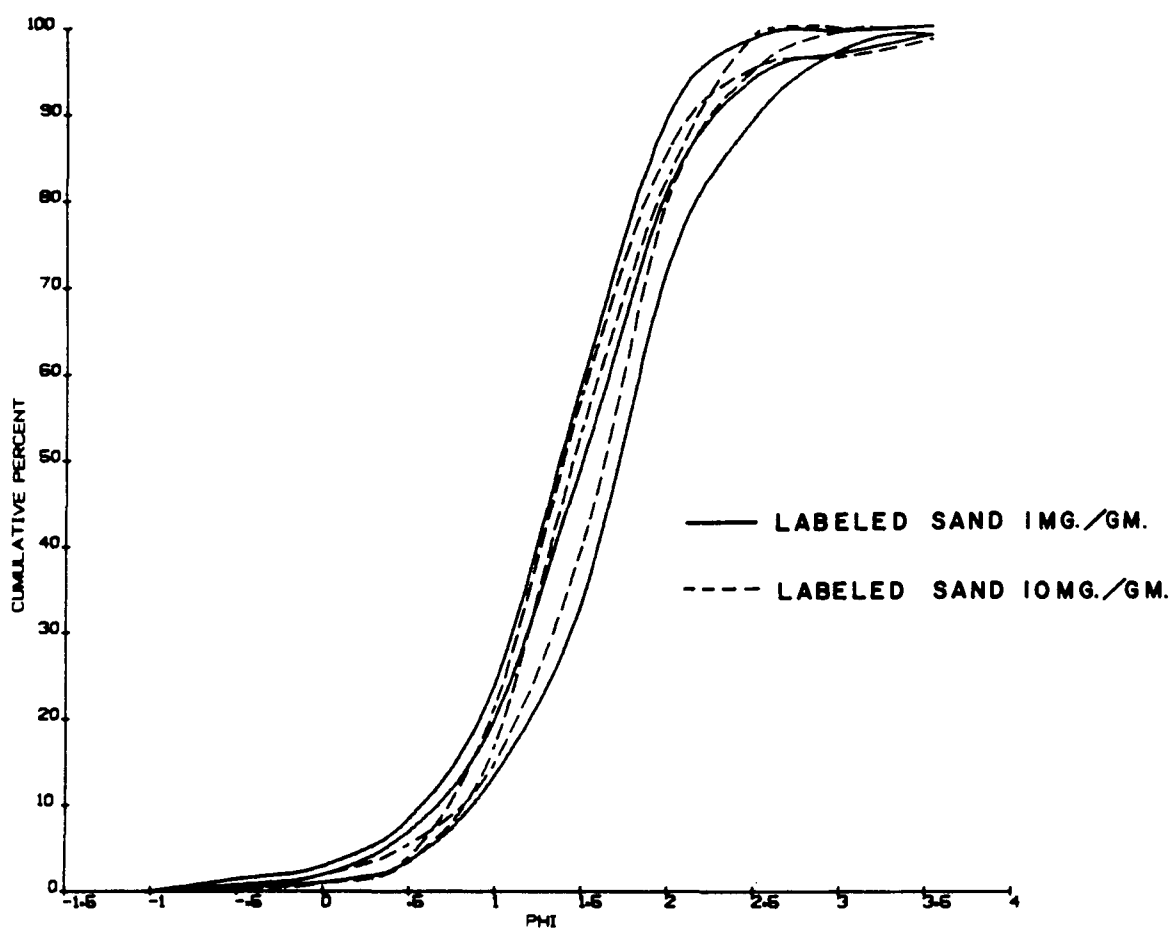
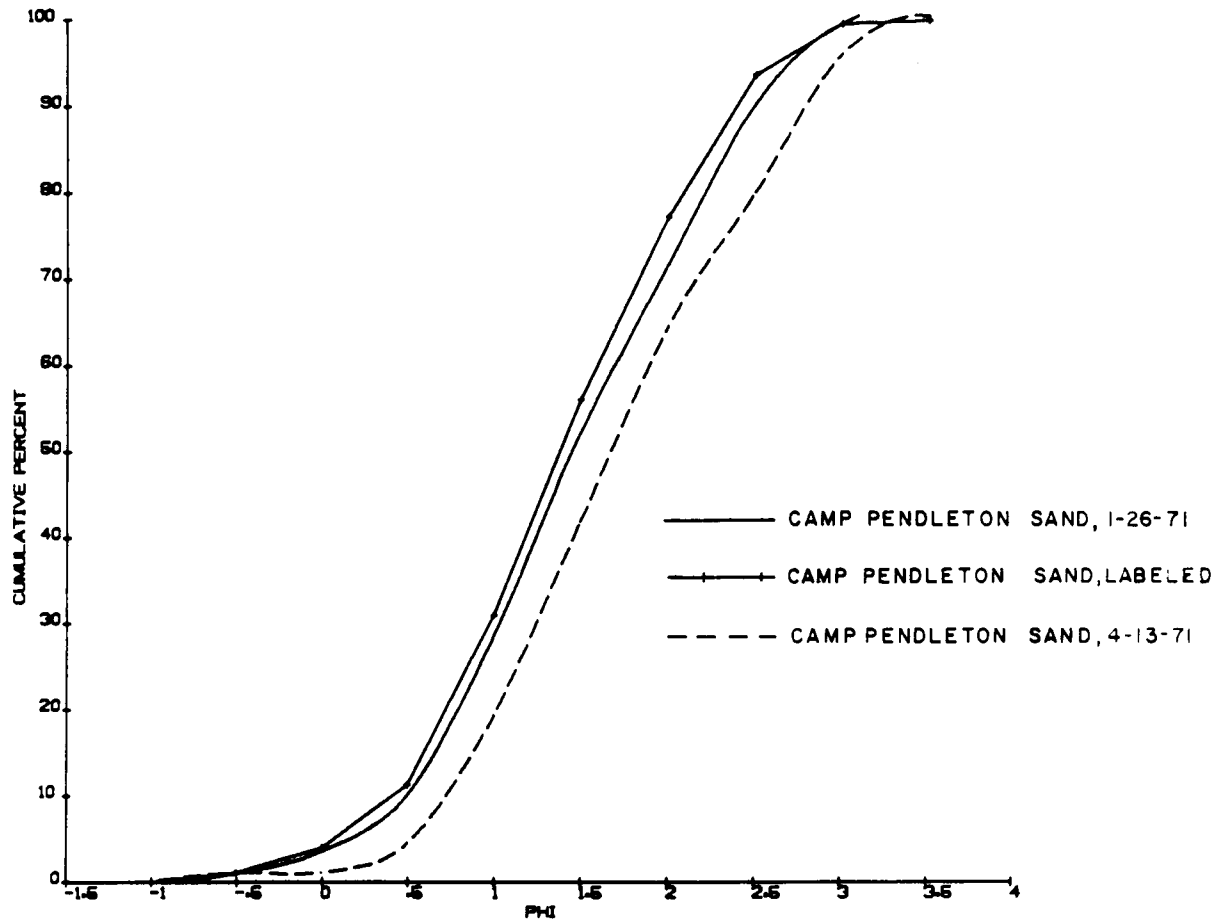


Fig. 15 Dry sieve analysis comparison of Camp  
Pendleton beach sand before labeling, after  
labeling and natural beach sand at the study  
area the day of the tracer study



both difficult and unreliable. Presumably, this unusually high background can be traced to the sediment load of the James, York and Rappahannock Rivers, all of which cut through a monazite belt which originates near Fredricksburg, Virginia and continues southwesterly to North Carolina.

Using the conversion chart (Fig. 18), obtained by plotting  $A_0$  values of various concentrations of dysprosium on sand, the weight percent or concentration of labeled sand in the study area can be determined.  $A_0$  values greater than 800 cpm are considered to be significant since this appears to be twice as much activity as could be expected from the natural occurrence of the rare-earth elements in the samples. An  $A_0$  value of 800 cpm corresponds to a weight percent of 0.0045 or 4.5 labeled grains per 100,000. This value corresponds to the probable lower detection limit of the stable isotope method and compares favorably with other previously described methods.

The dispersal patterns (Figs. 16 and 17) display several interesting features. The first set of samples taken indicate very high concentrations of tracer material along line C, which contains the two points of injection. After one hour, the tracer material apparently moved away from the injection points in both upcurrent and downcurrent directions. With a few localized exceptions, the remaining portion of the control grid was found to contain only negligible concentrations of labeled sand, less than 0.002%.

At the time of the second series of samples, 4 hours after injection, it appeared that a large mass of tracer sand had moved to lines F, G and H; the greatest concentrations appeared along line G. Mixing and burial are thought to account for the rather uniform distribution of low concen-

Fig. 16 Tracer dispersal pattern obtained 1 hr after injection at locations within rectangle. Data points and contours represent  $A_0$  values corrected to eliminate background activity

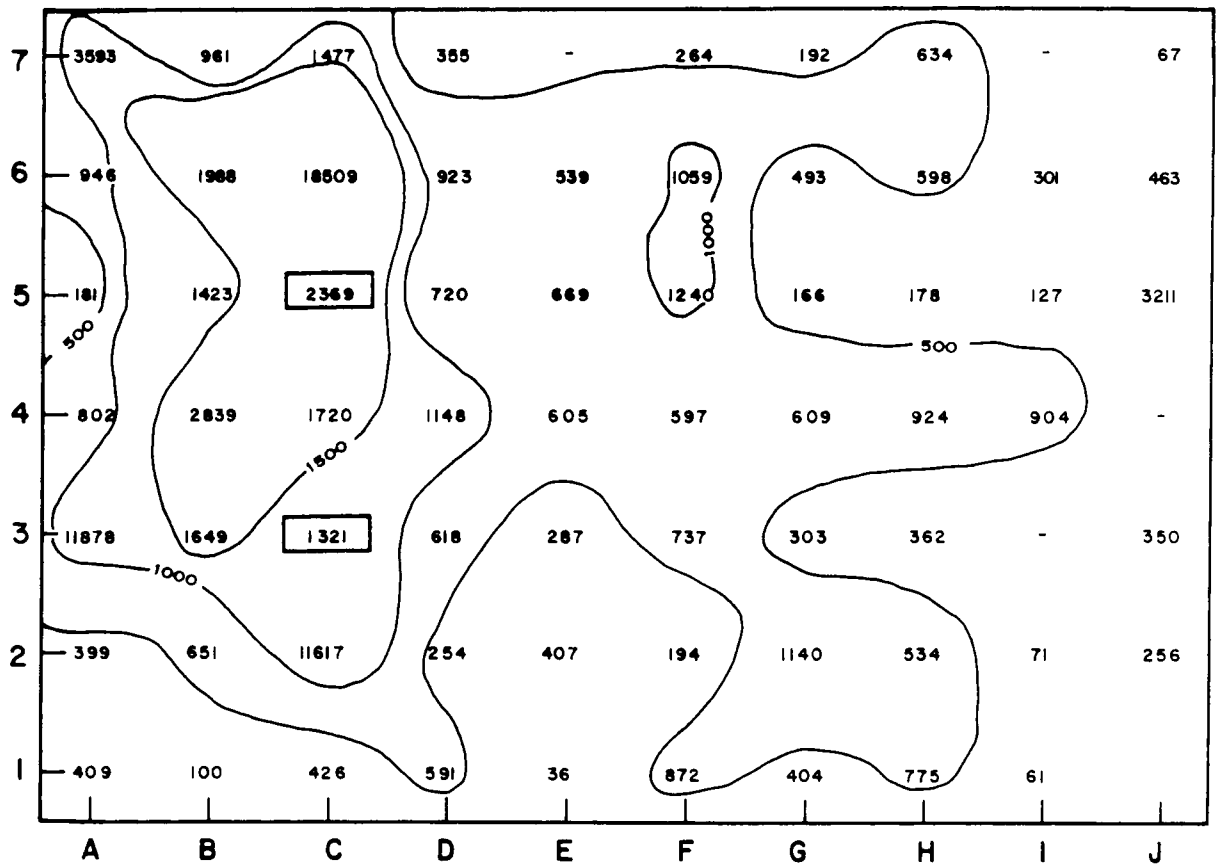




Fig. 17 Tracer dispersal pattern obtained 4 hrs after injection at locations within rectangle. Data points and contours represent  $A_0$  values corrected to eliminate background activity

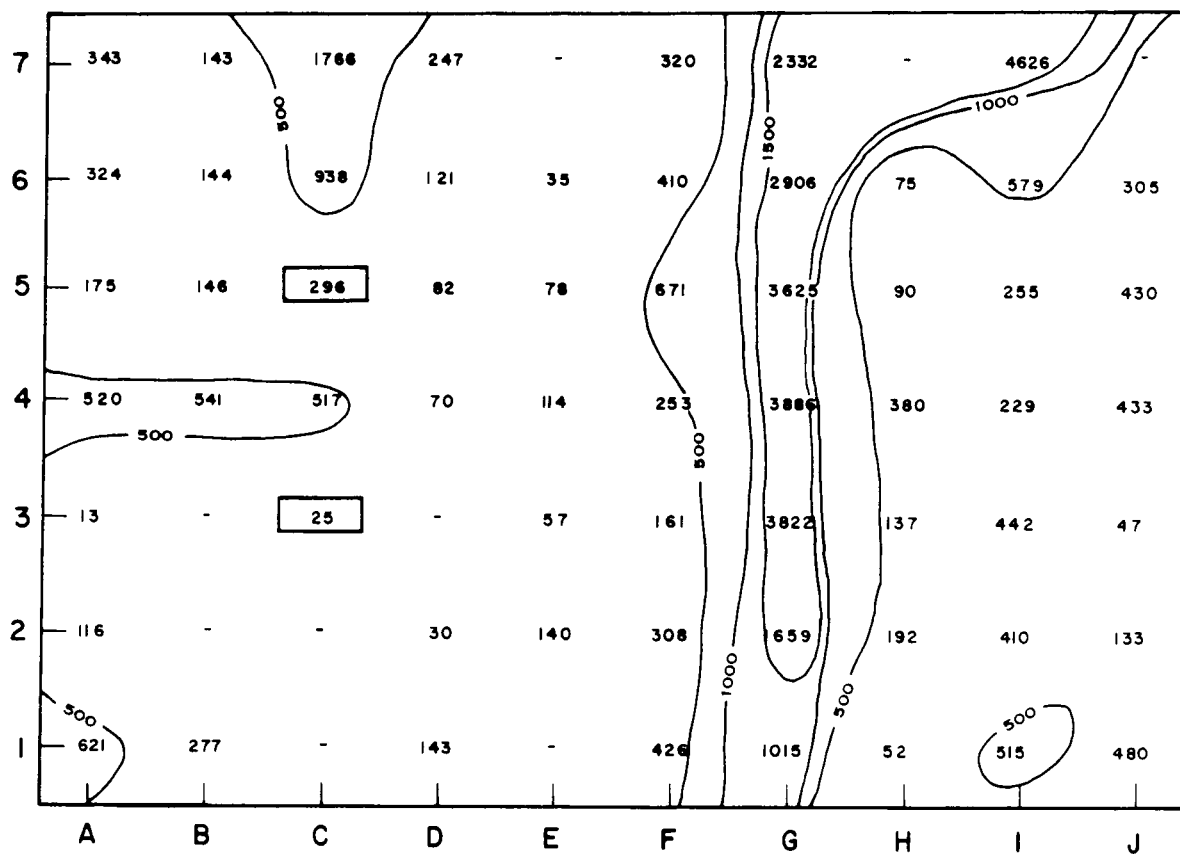
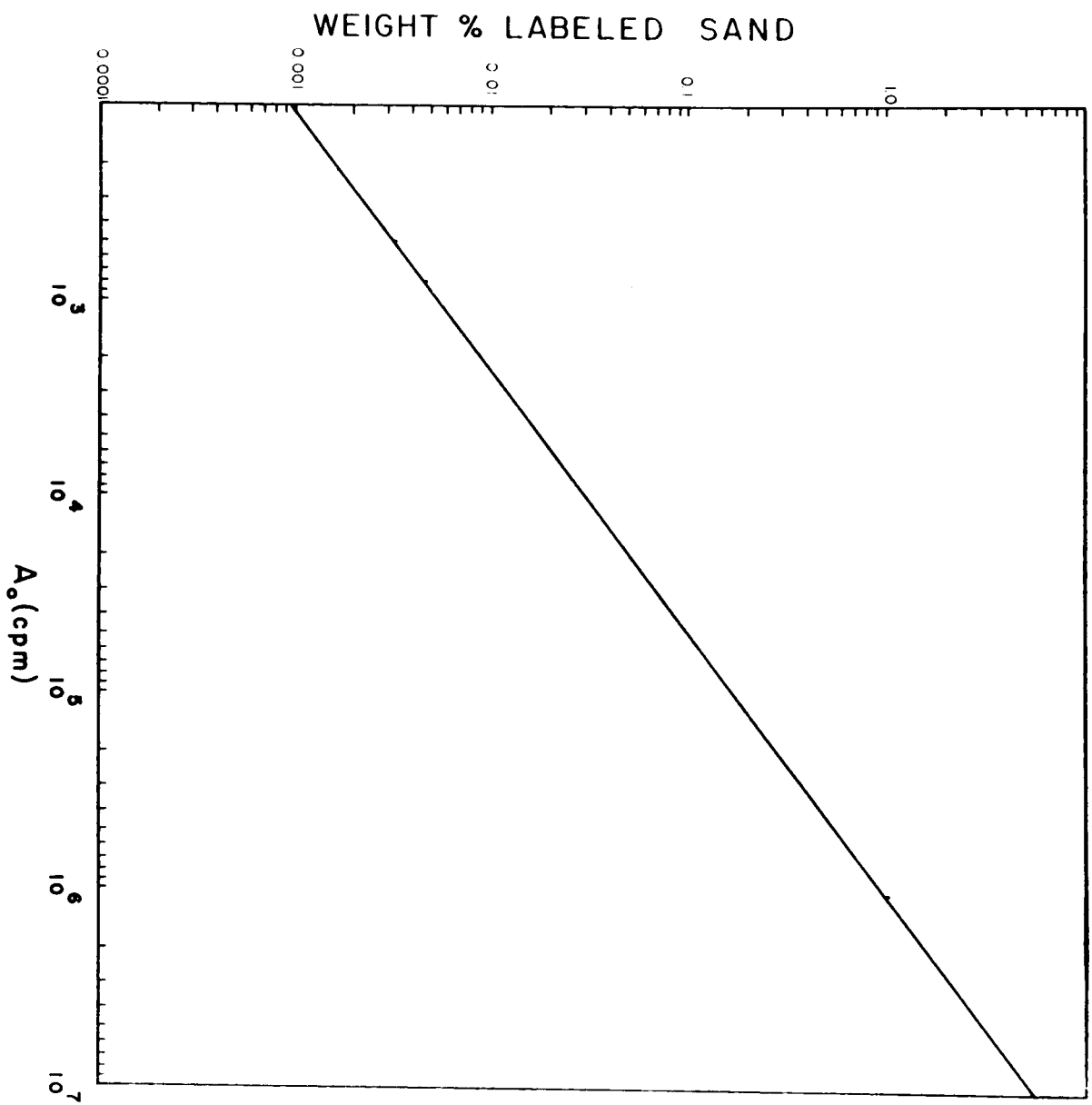


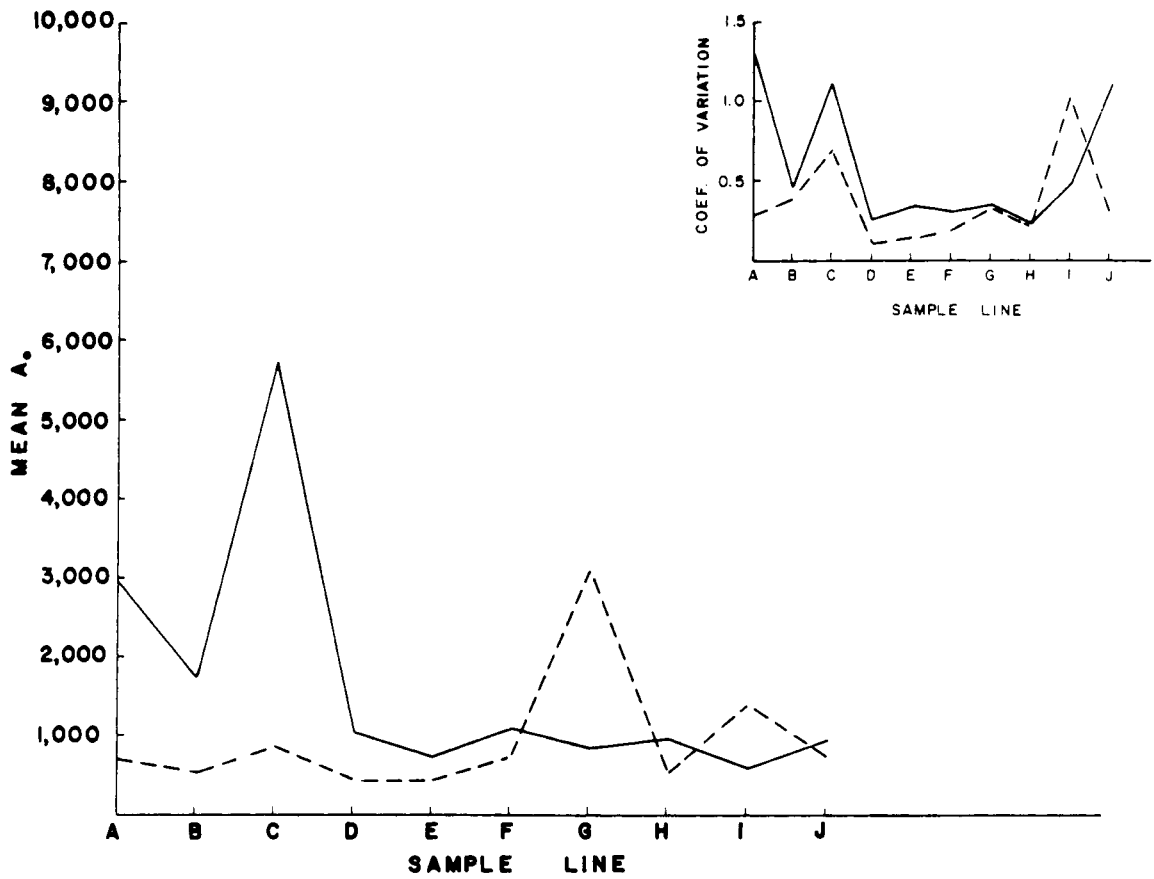
Fig. 18 Conversion chart used to change  $A_0$  values  
to weight percent labeled sand



trations of tracer over the remaining sections of the control grid. After 4 hours there did not appear to be a significant amount of tracer sand in the vicinity of the injection points. It is obvious that some of the labeled sand still remained within the control grid, evidence that 8.5 kg of tracer sand was sufficient to qualitatively study sediment transport over an area of  $3.5 \times 10^3 \text{ m}^2$  for time up to 4 hours. Wave and current conditions, listed in Table 5, remained rather uniform during the course of the field experiment. A mean tracer particle velocity of 0.42 ft/min was observed under the above conditions.

Mean  $A_0$  values for each sample line are plotted as a function of distance from tracer injection points in Fig. 19. Coefficients of variation for the same set of data are also shown. This coefficient is obtained by dividing the standard deviation by the mean. Unusually high coefficients (greater than 1.0), which indicate a large dispersion of data points about the mean, are found to occur at line A, C and J for the first set of samples. These high coefficients would tend to cause some question as to the validity of the two peaks. The wide range between the data points along lines A, C and J explain the high variations. Peaks at A and J may or may not be real, whereas the peak at C is felt to be real and representative of the recovery of some tracer sand near the injection points C-3 and C-5. Coefficients of variation for the second set of data taken 4 hours after tracer injection are relatively uniform, with an exception at line I. This high coefficient is explained by the present of one value (I-7, sample No. 273) which is more than 5 times greater than the remaining six samples along line I. Presumably this anomaly is due to some type of sample contamination prior to activation.

Fig. 19 Mean  $A_0$  values plotted against sample line. Coefficient of variation (standard deviation divided by the mean) is also plotted as a function of sample line. Solid line 1 hr past tracer injection; broken line 4 hr past tracer injection.



## CONCLUSIONS

1. The physical and chemical properties of the stable isotope tracer particles, as indicated by the abrasion and leaching tests, enable them to be used in sedimentary studies. Separate tests with samarium and lanthanum oxides indicated that no more than about 5% of the rare-earth oxide coating was lost from the sand grains through the combined effects of leaching and abrasion.
2. The refractory oxide coating, though stable to abrasion and seawater leaching, was acid soluble and can be quantitatively removed from the sand matrix through the stripping and concentration procedures developed.
3. The degree to which the tracer grains duplicated natural sand grains was evaluated using both RSA and dry sieving methods. Results seem to indicate that the coating level employed (10 mg/gm) does not significantly alter either the settling velocity or the effective diameter of the grain. Complete confidence in these results is somewhat lacking though due to the nature of both analytical techniques.
4. The additional weight imparted to one sand grain using the coating ratio of 10 mg per gram of sand is 0.6 $\mu$ g. This calculation is based on the assumption that the density of sand is 2.65 g/cm<sup>3</sup> and that the median diameter of the grain is 0.35 mm. This weight increase should not account for any significant alteration of the surface properties of the tracer particles.
5. The dispersal patterns obtained after 1 and 4 hours indicate that rare-earth labeled sedimentary particles can be followed after injection and dilution in the field. A mean particle drift rate of 0.42 ft/min was determined from the dispersal patterns.



6. Qualitative evaluation of sediment transport can only be achieved through the use of the acid stripped fraction of the field samples. The raw sand fraction was found to contain too much naturally occurring background dysprosium to be analyzed instrumentally. Beach and near-shore sand deposits in the Chesapeake Bay area were found to be very rich in rare-earth phosphate heavy minerals.
7. Because of the uncertainty of the true depth of the mobile layer of sand, no attempt was made to quantitatively determine the volume of sediment transported during the field test.
8. The absolute detection limit of the stable isotope method is between 4 and 5 grains per 100,000. After dilution of the tracer in the field, a more realistic detection limit was found to be about 1 grain per 10,000.
9. Preparation of 1 kg of stable isotope tracer sand, which requires about 5 hours, costs approximately \$10.00. Laboratory preparation of 100 samples for activation requires approximately 20 hours. Activation and counting of 100 samples requires an additional 5 hours. Reactor cost is approximately \$15.00 per hour. A total of 40 man-hours are required to prepare 1 kg of tracer sand, followed by the lab preparation and counting of 100 samples. The total cost involved using these figures and an arbitrary rate of \$2.00 per hour would be \$165.00 per kg of tracer sand prepared.
10. In light of the absolute detection limit obtained and the results of the field experiment, it is felt that the method evaluated can be justified. The stable isotope method can be justified and considered competitive with other methods since the detection limit obtained was

equal to or greater than fluorescent or radioisotope techniques.

11. Future refinements of the method would include the selection of either another rare-earth element or another element which could be similarly affixed to the sand particles. This element would not be a constituent of beach sand and, as a result, would not interfere with subsequent activation analysis. This refinement would ultimately lead to a less time consuming, more efficient, sample preparation and analysis system. This system would take full advantage of the neutron activation potential, thereby significantly increasing the tracer detection limit by a factor of at least ten.

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Table 1 Labeled Sand Abrasion Results

Sample	$\bar{x}$ counts less background	$\bar{x}$ counts abrasion filtrate	% tracer loss
La-140 labeled sand	27900	1171	4.1
Sm-153 labeled sand	3440	159	4.6

Table 2 Seawater leaching results

Sample	$\bar{\gamma}$ counts sand less background	$\bar{\gamma}$ counts leaching filtrate	% tracer loss
La-140 sand			
Sample 1	23924	92	0.30
Sample 2	22928	123	0.50
Sample 3	26722	103	0.30
Sm-153 sand			
Sample 1	2044	124	6.06
Sample 2	2174	124	5.70
Sample 3	2137	122	5.70

Table 3 Acid stripping and fluoride precipitation results

Sample	$\bar{x}$ counts sand less background	$\bar{x}$ counts stripped sand	$\bar{x}$ counts F <sup>-</sup> precipitate	% recovery
La-140 sand				
Sample 1	2090	15	2075	99.20
Sample 2	1926	33	1893	98.20
Sm-153 sand				
Sample 1	2229	182	2047	91.80
Sample 2	2218	172	2046	92.20
Sample 3	2172	184	1988	91.50
Sample 4	2193	167	2026	92.30
Sample 5	2085	171	1914	91.70



Table 4 Tabulated field data and corrected  $A_0$  values.

Sample No.	Sample ID	Corrected $A_0$ (cpm)	Sample No.	Sample ID	Corrected $A_0$ (cpm)
1	SAA11	1994	141	STA11	809
2	SAA21	2674	142	STA21	799
3	SAA31	2456	143	STA31	12278
4	SAA41	2262	144	STA41	1202
5	SAA51	2720	145	STA51	581
6	SAA61	3198	146	STA61	1346
7	SAA71	2728	147	STA71	3993
8	SAB11	3821	148	STB11	500
9	SAB21	1697	149	STB21	1051
10	SAB31	5400	150	STB31	2049
11	SAB41	2633	151	STB41	3239
12	SAB51	1978	152	STB51	1823
13	SAB61	2980	153	STB61	2388
14	SAB71	1974	154	STB71	1361
15	SAC11	2162	155	STC11	926
16	SAC21	2800	156	STC21	12017
17	SAC31	4230	157	STC31	1721
18	SAC41	4105	158	STC41	2120
19	SAC51	8240	159	STC51	2769
20	SAC61	3182	160	STC61	18909
21	SAC71	2970	161	STC71	1677
22	SAD11	2050	162	STD11	991
23	SAD21	3392	163	STD21	1654
24	SAD31	2628	164	STD31	1018
25	SAD41	3331	165	STD41	1548
26	SAD51	6218	166	STD51	1120
27	SAD61	3673	167	STD61	1322
28	SAD71	2336	168	STD71	755
29	SAE11	2502	169	STE11	436
30	SAE21	3582	170	STE21	807
31	SAE31	8270	171	STE31	687
32	SAE41	3814	172	STE41	1005
33	SAE51	4332	173	STE51	1069
34	SAE61	4035	174	STE61	939
35	SAE71	3541	175	STE71	347
36	SAF11	1980	176	STF11	1272
37	SAF21	4133	177	STF21	594
38	SAF31	4073	178	STF31	137
39	SAF41	4206	179	STF41	997
40	SAF51	4001	180	STF51	1640
41	SAF61	4841	181	STF61	1459
42	SAF71	3273	182	STF71	664
43	SAG11	3263	183	STG11	804
44	SAG21	3993	184	STG21	1540
45	SAG31	2793	185	STF31	703
46	SAG41	3200	186	STG41	1009
47	SAG51	3193	187	STG51	566
48	SAG61	2905	188	STG61	893
49	SAG71	2565	189	STG71	592

Table 4 Continued

Sample No.	Sample ID	Corrected A <sub>o</sub> (cpm)	Sample No.	Sample ID	Corrected A <sub>o</sub> (cpm)
50	SAH11	3358	190	STH11	1175
51	SAH21	2803	191	STH21	934
52	SAH31	1973	192	STH31	762
53	SAH41	984	193	STH41	1324
54	SAH51	3539	194	STH51	578
55	SAH61	3618	195	STH51	998
56	SAH71	3332	196	STH71	1037
57	SAI11	1788	197	STI11	461
58	SAI21	2172	198	STI21	471
59	SAI31	3882	199	STI31	397
60	SAI41	2207	200	STI41	1304
61	SAI51	1705	201	STI51	527
62	SAI61	2352	202	STI61	701
63	SAI71	2196	203	STI71	883
64	SAJ11	1888	204	STJ11	458
65	SAJ21	3071	205	STJ21	656
66	SAJ31	2444	206	STJ31	750
67	SAJ41	-	207	STJ41	-
68	SAJ51	1768	208	STJ51	3611
69	SAJ61	2368	209	STJ61	863
70	SAJ71	2462	210	STJ71	467
71	SAA12	1960	211	STA12	1021
72	SAA22	2639	212	STA22	516
73	SAA32	2839	213	STA32	413
74	SAA42	2453	214	STA42	920
75	SAA52	3057	215	STA52	575
76	SAA62	3342	216	STA62	724
77	SAA72	2228	217	STA72	743
78	SAB12	2176	218	STB12	677
79	SAB22	3282	219	STB22	267
80	SAB32	5698	220	STB32	302
81	SAB42	5494	221	STB42	941
82	SAB52	2862	222	STB52	546
83	SAB62	2854	223	STB62	544
84	SAB72	3988	224	STB72	543
85	SAC12	2556	225	STC12	374
86	SAC22	2676	226	STC22	272
87	SAC32	4961	227	STC32	425
88	SAC42	5144	228	STC42	914
89	SAC52	3621	229	STC52	696
90	SAC62	4136	230	STC62	1338
91	SAC72	2808	231	STC72	2166
92	SAD12	1955	232	STD12	543
93	SAD22	4857	233	STD22	430
94	SAD32	4364	234	STD32	396
95	SAD42	2955	235	STD42	470
96	SAD52	5902	236	STD52	482
97	SAD62	2995	237	STD62	521
98	SAD72	3755	238	STD72	647

Table 4 Continued

Sample No.	Sample ID	Corrected $A_0$ (cpm)	Sample No.	Sample ID	Corrected $A_0$ (cpm)
99	SAE12	3967	239	STE12	316
100	SAE22	2795	240	STE22	540
101	SAE32	3280	241	STE32	457
102	SAE42	3220	242	STE42	514
103	SAE52	3480	243	STE52	478
104	SAE62	3175	244	STE62	435
105	SAE72	1960	245	STE72	385
106	SAF12	3683	246	STF12	826
107	SAF22	3227	246	STF22	708
108	SAF32	5466	248	STF32	561
109	SAF42	3300	249	STF42	653
110	SAF52	4230	250	STF52	1071
111	SAF62	5088	251	STF62	810
112	SAF72	2372	252	STF72	720
113	SAG12	5037	253	STG12	1415
114	SAG22	3685	254	STG22	2059
115	SAG32	5062	255	STG32	4222
116	SAG42	4293	256	STG42	4286
117	SAG52	3456	257	STG52	4025
118	SAG62	4662	258	STG62	3306
119	SAG72	3993	259	STG72	2732
120	SAH12	1933	260	STH12	452
121	SAH22	4146	261	STH22	592
122	SAH32	3853	262	STH32	537
123	SAH42	3853	263	STH42	780
124	SAH52	2546	264	STH52	490
125	SAH62	4580	265	STH62	475
126	SAH72	2400	266	STH72	315
127	SAI12	3050	267	STI12	915
128	SAI22	4414	268	STI22	810
129	SAI32	5285	269	STI32	842
130	SAI42	4664	270	STI42	629
131	SAI52	4614	271	STI52	655
132	SAI62	3828	272	STI62	979
133	SAI72	2221	273	STI72	5026
134	SAJ12	1752	274	STJ12	880
135	SAJ22	3296	275	STJ22	533
136	SAJ32	5976	276	STJ32	447
137	SAJ42	3992	277	STJ42	833
138	SAJ52	2408	278	STJ52	830
139	SAJ62	3144	279	STJ62	705
140	SAJ72	4320	280	STJ72	369

Table 5 Physical and meteorological parameters  
during tracer study

Time (hrs)	Wave height $H_b$ (m)	Period (sec)	Wave direction	Longshore current direction	Longshore current velocity (kn)	Wind direction and velocity (kn)
0930	0.45	5	SE	North	0.49*	South 6
1035	0.61	5	SE	North	0.15	South 9
1230	0.45	4	SE	North	0.15	SSE 7
1330	0.30	5	SE	North	0.20	SSE 7
1430	0.61	5	ESE	North	0.20	SSE 0.08
1530	0.45	7	ESE	North	0.30	SSW 11

\* Measured on surface with a grapefruit