

DESIGN AND USE OF A
FIELD ACTIVATION ASSEMBLY

A THESIS

Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
in partial fulfillment of the
requirements for the degree of
Master of Science

in

The Department of Nuclear Engineering

by
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B.S., Louisiana State University, 1971
December, 1973

ACKNOWLEDGEMENT

The author would like to acknowledge the following people for their assistance in this research. Dr. Frank A. Iddings for his help and understanding throughout my graduate career. Dr. Robert C. McIlhenny and Dr. John C. Courtney for their help and time whenever it was needed. Calvin E. Pepper and Lisle R. Posey for their help during the construction of the laboratory and field apparatus.

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ABSTRACT

The use of thermal neutron activation analysis for cement content analysis of soil cement samples should provide a more reliable roadbed surface or foundation in parts of Louisiana where the soil base is structurally weak. The design of a field radiation shield-activation assembly should be based on portability and reliability, biological shielding and predictable and reproducible irradiation geometry requirements. A spherical activation shield assembly has been constructed to hold a 200 microgram californium-252 source. This assembly has allowed cement content analysis to a precision of $\pm 5\%$ (of cement added). This system has provided the necessary thermal neutron flux and biological shielding required for the analysis of over 700 samples in the laboratory or field environments.

CHAPTER ONE

Introduction

Over the past two decades, there has been a significant increase in the importance of testing construction materials to provide reliable estimates of their lifetimes and physical properties. Greater reliability is essential, as the cost of repair and reconstruction can exceed the original construction costs. Through the use of either non-destructive or destructive testing techniques, a contractor or engineer can be assured that the finished product meets contract specifications. The physical properties of many highway construction materials can vary greatly with their composition. For example, the physical properties of concrete or soil cement can vary greatly with small changes in moisture, aggregate, or cement contents.

In many parts of Louisiana, the lack of a good, firm soil base makes the construction of roadbeds difficult. The generally soft soil base, made up of clay, alluvial, and marsh soils, requires stabilization before a roadbed can be constructed. Stabilization is accomplished through two basic methods: lime stabilized soils and soil cements. Lime stabilized soils are produced by mixing from just above zero to ten percent lime (by weight) with the soil immediately below the surface of the roadbed. More often used, soil cements are a mixture of from one to seventeen percent (by weight) of cement (usually portland cement) with either the roadbed or the road surface itself.

The quality of roadbed construction at this time has been dependent upon the ability of contractors to obtain the proper mixture of lime or

cement with soil. This problem is caused by the manner in which these materials (soil and cement or lime) are mixed. Currently there are three major mixing processes. The first uses a large mixing machine that scoops up four to twelve inches of roadbed soil and automatically adds specified amounts of cement and water to the soil in the roadbed over which it travels. The second method involves laying and spreading a load of cement over the roadbed, which is then churned up; later water can be added by sprinkler trucks, and the mixture is then compacted. The third technique involves the use of a pug-mill to produce soil cement mixtures, which are then trucked to the construction site. It is during these various operations that differences in cement content (weight percent) can occur.

In order to provide a more consistent roadbed composition it is necessary to provide some method of testing the mixed soil cement for cement content before it hardens. The current methods of testing are titration, atomic absorption spectroscopy, X-ray fluorescence, and backscatter techniques. These tests usually determine the amount of calcium in the sample as an index of the cement content. The titration technique usually consists of dissolving one gram of the soil cement sample in Na_2O_2 or HF and then titrating the Ca^{+2} ion with ethylene diamine tetraacetic acid. The color indicator erichrome black T is used to determine when the titration end point is reached. The atomic absorption spectroscopy technique consists of dissolving the sample and then aspirating it into a flame through which is passed monochromatic light of the wavelength emitted by the excited atom in question; this light is then absorbed by the atoms in the flame and the amount of absorption is measured photometrically to establish the amount of

calcium present.

The X-ray fluorescence technique consists of exciting atoms in a sample with X-rays or beta particles and observing the characteristic X-rays of the sample that are emitted. Another technique also used to determine calcium content of wet mixes is the X-ray backscatter technique. It consists of observing the backscattered radiation from an Am^{241} and a Cs^{137} source (Am^{241} for the cement content and Cs^{137} for the density correction).

However, these techniques suffer from the following limitations. The first two (titration and atomic absorption spectroscopy) use very small samples, usually on the order of one gram. Therefore, the sample is not a true representation of the mixture as one gram of concrete or soil cement can vary greatly with another gram taken from the same area. A larger sample is needed to show a true average composition of the mixtures. The second two techniques (X-ray fluorescence and X-ray backscatter) suffer from a lack of penetration into the sample. Their effective depth of determination is in the small fraction of an inch range. Here, again, the sample composition determined is not necessarily representative of the total volume. All of the techniques described above require well equipped laboratories. Additionally, many hours of personnel training are required and the tests themselves are time consuming and tedious.

The research described in this thesis will show that neutron activation analysis of the soil cement samples is an accurate and fast technique for determining the cement content of two-to-three kilogram samples of soil cement. This technique, by virtue of its deeper penetration and ease of analysis, should fit the needs of a highway

department on-site testing facility. The shielded neutron source and the counting equipment necessary for analysis can be made field portable. The system can provide quantitative determination of cement content to within an accuracy of within five percent of the absolute amount of cement added. Data are available within ten to twenty minutes so that deficiencies can be brought to the attention of the contractor while the work is in progress.

Activation analysis consists of inducing radioactivity in a specimen containing stable atoms for the purpose of qualitative and/or quantitative assay. Thermal neutron activation of a sample is accomplished by placing the sample in a minimal-energy neutron flux (ϕ) for a specified period of time. These neutrons are captured by the nuclei of a variety of atoms in the sample. This capture by a stable atom results in a simple nuclear transformation which may result in the formation of a radioactive species that emits radiation (electromagnetic and/or particulate) amenable to detection. The amount of activity (i.e., disintegration rate) that can be produced in a sample is expressed by:

$$A = N\phi\sigma \left[1 - e^{(-0.693 t_a/t_{1/2})} \right] \left[e^{(-0.693 t_d/t_{1/2})} \right] \quad (\text{Eq. 1-1})$$

where

$N\phi\sigma$ = rate of production

$1 - e^{(-0.693 t_a/t_{1/2})}$ = saturation factor

$e^{(-0.693 t_d/t_{1/2})}$ = decay factor

and

Λ = induced activity (d/s)

N = number of target atoms

ϕ = neutron flux (n/cm² - sec)

σ = capture cross section ($\times 10^{-24}$ cm²)

$t_{1/2}$ = half life of resultant isotope

t_a = irradiation time

t_d = decay time

The neutron capture cross section is the probability of neutron capture and is an energy-dependent variable. Identifying the isotope of interest can be done chemically or by nuclear counting procedures. Chemical identification is long and tedious and requires trained technicians. However, radioactive isotopes can be identified by observing the particular types and energies of emission. Gamma spectroscopy is a very useful tool in the determination of various gamma ray energy emitters. Using gamma spectroscopy the specific energy(ies) of emission can be counted and analyzed. This analysis is done electronically by measuring emissions versus their energies.

In this study the particular isotope of interest, Ca⁴⁹, has gamma energies of 3.08 and 4.05 MeV which are much higher than the other energies of gamma rays emitted by the isotopes found by activation of soil and soil cement. This can be seen in Table 1-1.

The high energies of gamma rays and short half-life of Ca⁴⁹ reduce the possibility of interference to the activation method of cement content analysis. Figure 1-1 is a typical gamma spectrum obtained from the activation of a soil cement sample.

The final step in activation analysis consists of the quantitative

Table 1-1

<u>Element</u>	<u>Activation Product</u>	<u>Cross Section* (Thermal capture)</u>	<u>Product Half-life</u>	<u>Gamma Energy (MeV)</u>
Aluminum	Ad ²⁸	0.234 b	2.3 min.	1.78
Calcium	Ca ⁴⁷	1.2 b	4.7 days	0.81, 1.30
Calcium	Ca ⁴⁹	0.2 b	8.8 min.	3.08, 4.05
Iron	Fe ⁵⁹	1.2 b	45 days	1.09, 1.29
Manganese	Mn ⁵⁶	13.3 b	2.58 hrs.	0.84, 1.81
Potassium	K ⁴²	1.5 b	12.4 hrs.	1.52
Scandium	Sc ⁴⁶	25 b	85 days	0.89, 1.12
Silicon	Ad ²⁸	0.16 b**	2.3 min.	1.78
Sodium	Na ²⁴	0.53 b	14.8 hrs.	1.37, 2.25

*fast neutron cross section, (n, p)

**1 b (barn) = 10^{-24} cm^2

assay. With a single isotope or an isotope with a particular energy gamma ray emission, the number of original target atoms can be determined using equation 1-1. However, this method is limited by the inaccuracies in ϕ , σ , t_a , $t_{1/2}$, t_d . Even when all timing problems are solved, ϕ and σ due to their energy dependence can only be estimated.

The activation analysis technique used in this study will be to prepare standard samples of known cement content and perform activation analysis on these samples to establish a calibration curve (see Figure 1-2). The use of a calibration curve avoids unnecessary and tedious absolute calibrations of the detector system for cement content analysis. The calibration curve also allows quick, on the spot results in the field with as little chance of error as possible. The linear nature of the

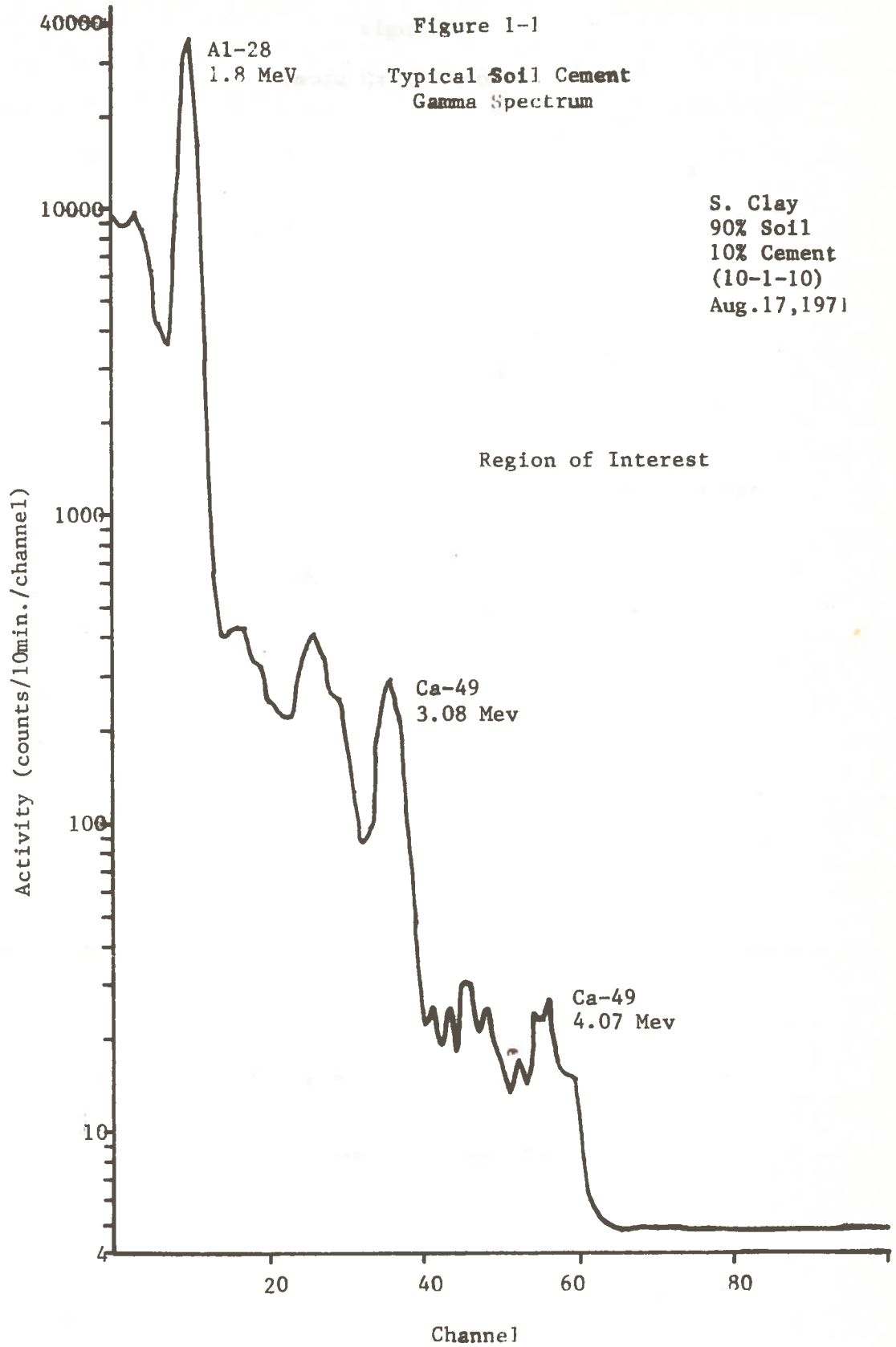
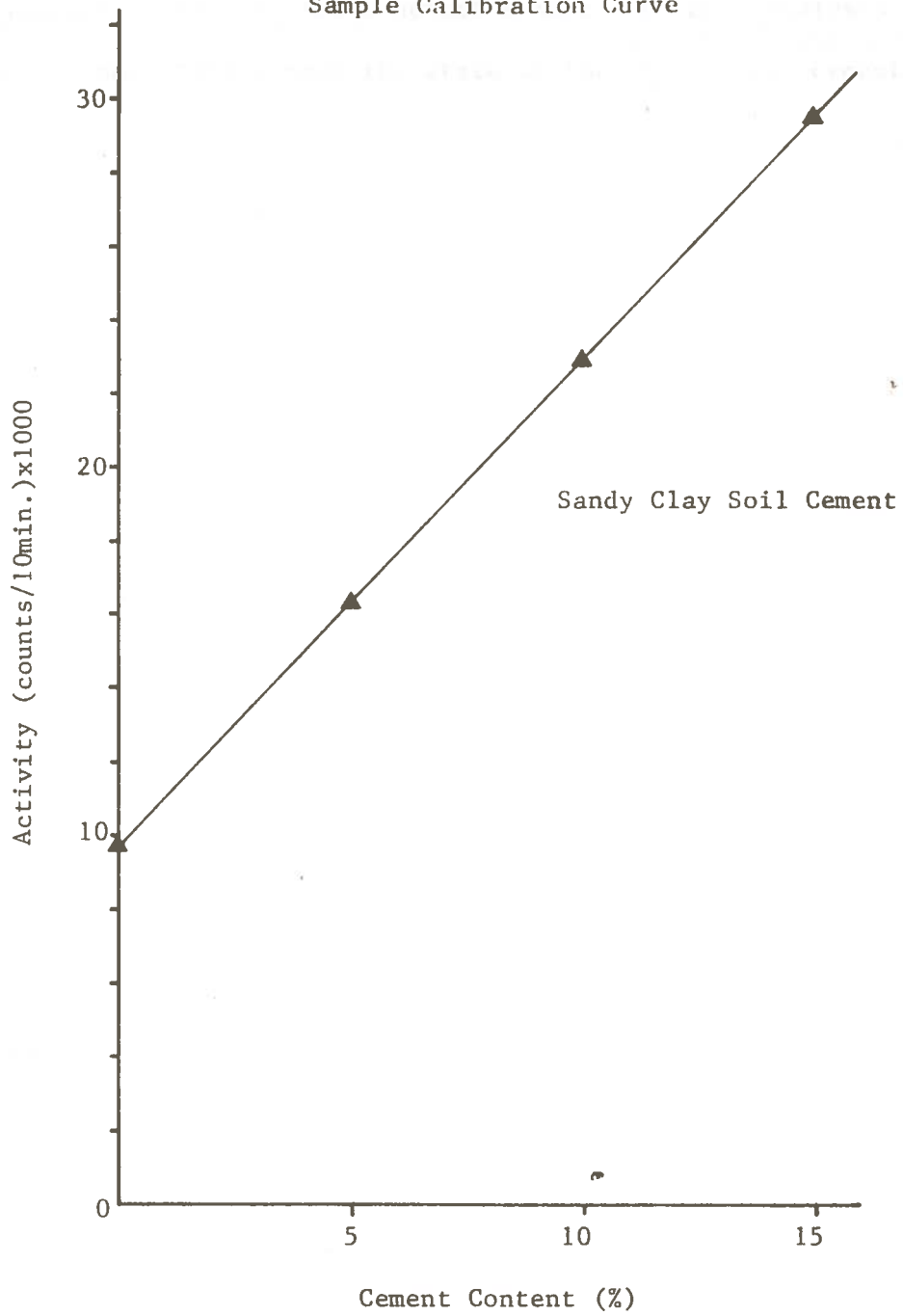


Figure 1-2

Sample Calibration Curve



curve then makes quantitative analysis of the unknown samples possible to within a few percent of the actual cement content. Analysis to this accuracy is much better than the state of the art of soil cement mixing.

CHAPTER TWO

Design Criteria

In order to provide a reliable activation analysis study in the highway construction site environment, five criteria must be met by the equipment. It must have:

1. A reliable and predictable source of neutrons.
2. A portable biological shield for the neutron source.
3. A portable constant geometry activation assembly.
4. A stable, reliable timing and counting system.
5. Portable field monitoring equipment for insuring radiological safety.

This study shall be devoted, primarily, to the fulfillment of the first three criteria above.

The choice of a neutron source must be based on the following criteria:

1. The source must be small and compact.
2. The source must be highly reliable.
3. The source must have a near constant or predictable flux output.
4. The biological shielding requirements of the source must be met.

Neutron generators (particle accelerators) provide a small compact neutron source that requires little or no shielding (other than distance) since they can be turned on or off. However, for field work these generators are not applicable due to their non-constant or unpredictable output (flux). They also require a power source for the high D. C. voltage necessary for operation. These generators are also expensive

(priced in excess of \$10,000 each).

Isotopic sources provide the necessary predictable flux and have high reliability. The relatively small size of the isotopic source is offset somewhat by the size of the biological shield that must accompany it. Among the isotopic sources there are three major problems (excluding cost): short half-lives, high heat generation and low emissivity.

Californium 252 was chosen as the neutron source for this study due to:

1. Its ready availability (L. S. U. Californium Demonstration Center).
2. Its low heat generation (the lowest of all isotopic neutron sources commercially available at present).
3. Its reasonable half-life (2.65 years).
4. Its high neutron emission rate (2.34×10^{12} n/sec - g).
5. Its small volume.

Californium-252 decays by both alpha emission and spontaneous fission. There is about one spontaneous fission for every 31 alpha particle emissions. Each fission also produces 3.76 neutrons emitted in a fission spectra having an average energy of about 2.3 MeV and a most probable energy of about 0.8 MeV. It also emits 1.3×10^{13} gamma photons per second per gram, while its heat generation is only 39 watts per gram.

The high emission rate of both neutrons and gamma photons by a Californium-252 source requires a biological shield. To provide protection for the technicians involved in activation analysis from radiation exposure not only must the source be shielded but the area around the shield must be monitored with suitable radiation detection equipment. Access to this area must also be restricted to monitored individuals.

To optimize the design of an Activation Shield Assembly, the following criteria must be considered: the system must be of a size such that it is field portable, it must provide sufficient neutron flux for activation analysis of soil cement samples, and it must provide a biological shield for the operators using it. The problems of constructing a field portable Activation Shield Assembly require consideration of total weight, modes of possible transportation, size (volume), and reliability. The total weight of the shield is dependent upon the size, shape, and material of the finished product. The volume relations for three of the simplest shapes to construct compare as follows:

$$\text{cube} \quad V = s^3$$

$$\text{cylinder} \quad V = 0.785s^3$$

$$\text{sphere} \quad V = 0.523s^3$$

where

V = total volume

s = diameter, height, length, or width

Therefore, it is readily seen that a spherical or an approximate spherical shape is the optimum with regards to weight. This weight savings must be compared with the amount of extra cost and time involved in constructing a spherical shield. The principal material used for construction is water-extended polyester (WEP) which is a mixture of 50% water and 50% Alpon 661-P resin^{1/} with hydrogen peroxide used as a catalyst or hardening agent. Water extended polyester was chosen because of its high hydrogen atom content. This high hydrogen concentration results in a high degree of moderation (slowing down of the fast neutrons emitted by the Cf²⁵² source) and its resulting better

¹A product of Ashland Chemical Company, Houston, Texas.

shielding properties for neutron radiation. WEP was chosen over other hydrogenous materials such as water, parafin, glycerin, or polyethylene because it can be used to form a safe reliable shield system at reasonable cost. It is fireproof, easy to shape by casting or wood working tools, and is not affected by ambient temperatures. Also there is no chance for the hydrogenous constituent to leak from the assembly.

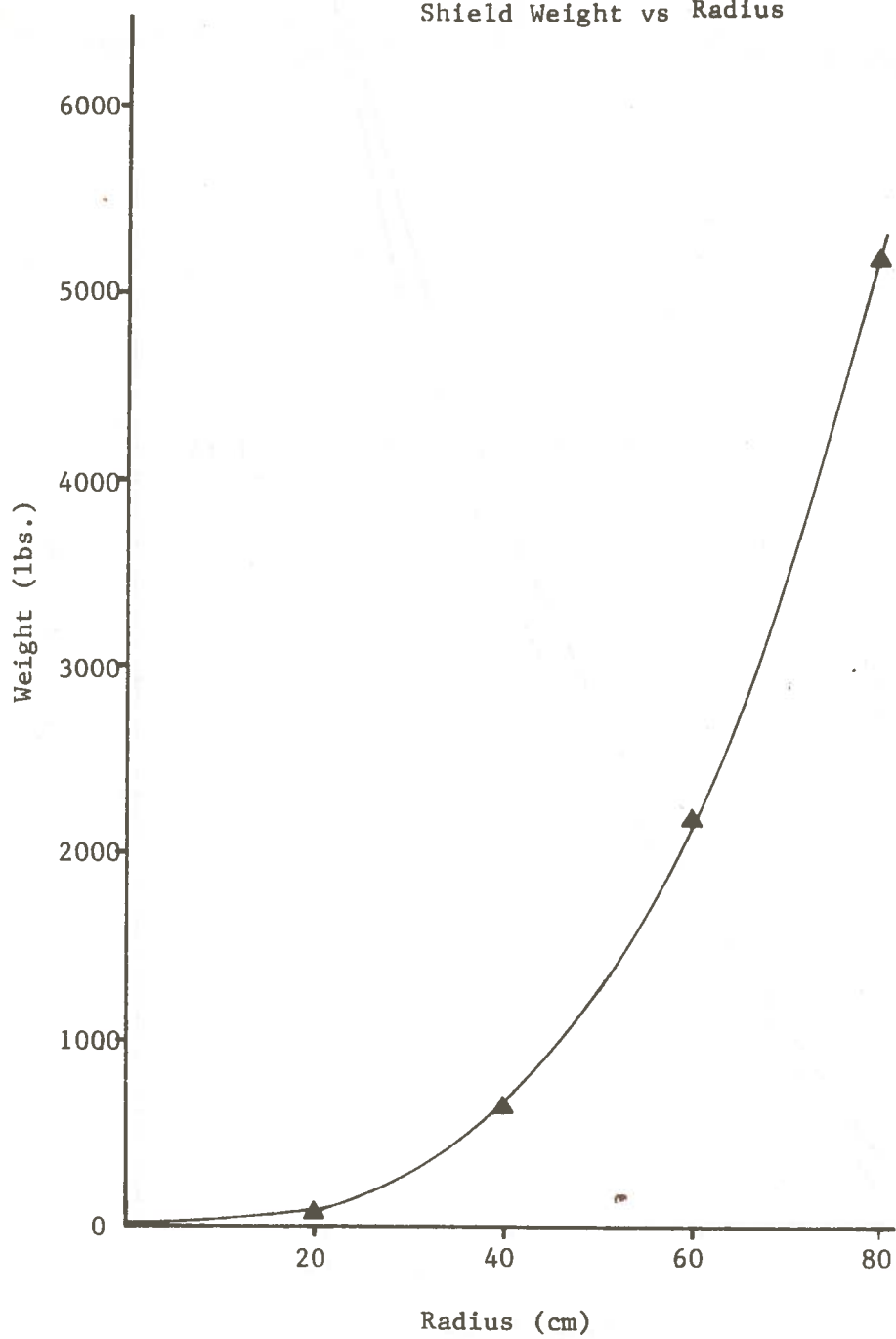
Water, glycerin, and parafin are fluids and must be contained in some sort of vessel. Water, the cheapest, has problems with regard to temperature variations of the environment (some action must be taken to prevent freezing during winter months). All liquid shields run the risk in a field environment of leakage leaving an essentially bare source at a construction site. Solid polyethylene has many advantages but it is very expensive and hard to machine.

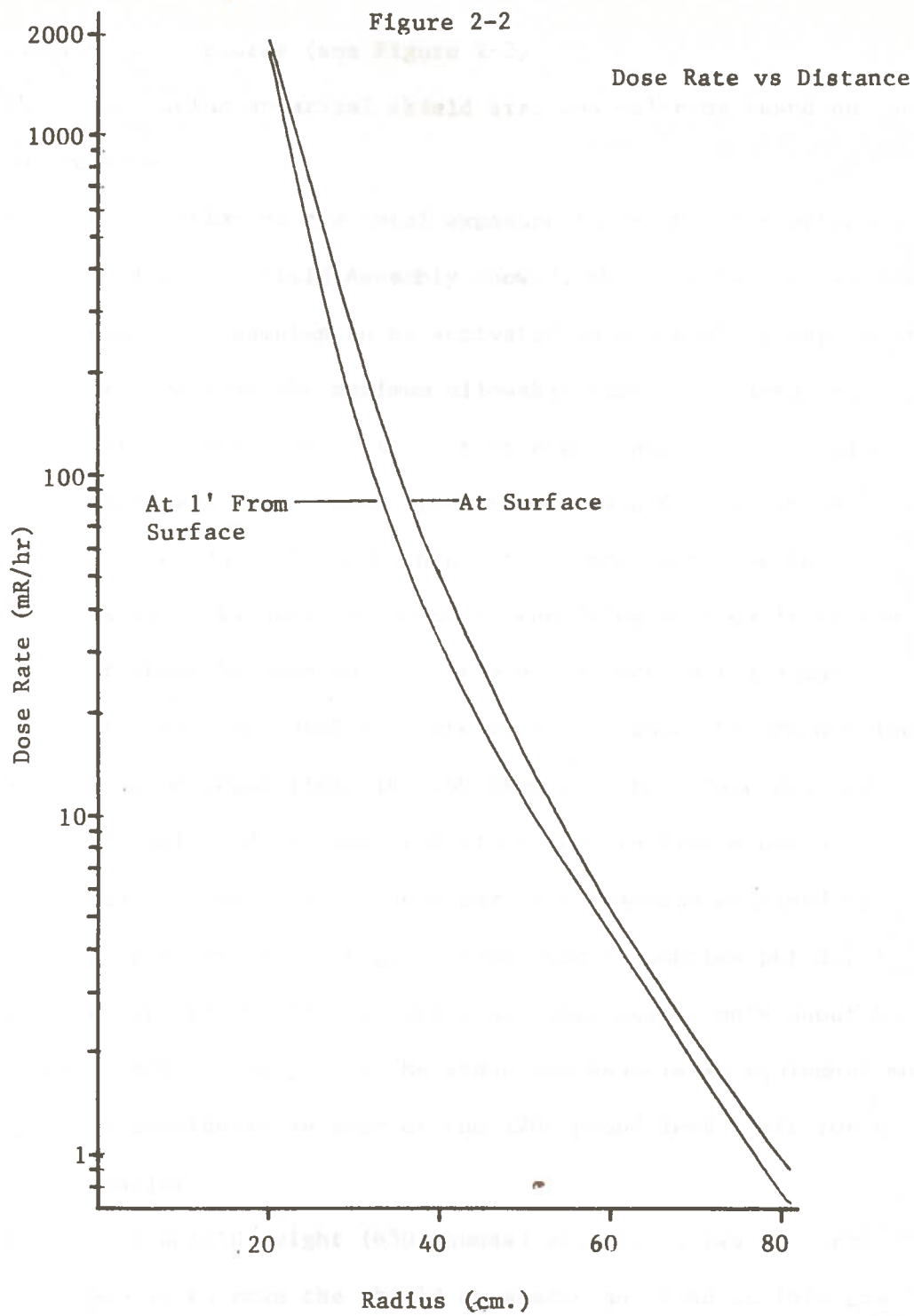
The total weight of a field portable activation shield assembly must be kept to under 1200 pounds (the axle rating on most readily available rental trailers). The relationship of radius to weight for a spherical shield can be seen in Figure 2-1.

The shielding requirements will be determined by the size (mg) of the Cf²⁵² source to be used, and the maximum allowable radiation exposure to operators. The source size required is between 100 and 200 micrograms of Cf²⁵². The exposure rates to personnel using the assembly must not exceed 1.25 R/quarter (the maximum legal exposure to monitored individuals). This figure is a maximum and every effort should be made to reduce the exposure to the lowest practical level. The following data was calculated^{2/} for various sizes of shields using a worst case of a

^{2/} Californium Progress, Vol. 7, April 1971, published by U.S.A.E.C Savannah River, p. 40.

Figure 2-1
Shield Weight vs Radius





200 microgram Cf²⁵² source (see Figure 2-2).

The 40 cm radius spherical shield size was selected based on the following reasons:

1. Calculation of the total exposure to an operator using the Activation Shield Assembly showed, that assuming a maximum number of samples to be activated in one working day, would be less than the maximum allowable exposure. Assuming a rate of ten samples an hour or eight samples in a full working day the total exposure time would be about 30 minutes/day. The exposure rate at one foot from the assembly is about 32 mRem/hr. resulting in a daily exposure of about 16 mRem/day. Over a 90 day period (calendar quarter) the total exposure would be about 1050 mRem. The maximum legal limit is 1250 mRem over this same period. (Actual studies conducted after construction shows a maximum sample run in one day of 40 samples analyzed by two people, and an average of less than 20 samples per day.)
2. While the shield activation assembly weighs only about 650 pounds the weight of the stand and associated equipment must be considered as part of the 1200 pound load limit for a trailer.
3. This shield weight (650 pounds) will allow two or three people to move the shield on wheels and load it into the trailer without the use of a fork lift or a crane which is expensive and would be awkward in a closed trailer.

The determination of source size and source-sample geometry will be discussed in a later chapter.

CHAPTER THREE

Preliminary Studies

The results of prior research showed that cement content analysis could be performed by activation analysis techniques using Ca^{49} as the isotope of interest.^{3/} Thermal neutron activation analysis was performed using reactor and isotopic (PuBe) neutron sources to provide the essential neutron flux. Fast neutron activation analysis was also performed using a linear particle accelerator (neutron generator) to provide the neutron flux. However, since it is the object of this research to produce a field-portable system with results obtained a few minutes after preparation of the samples from the roadbed, the technique of thermal neutron irradiation in a nuclear reactor was not acceptable. Neutron generators of small enough size for field use have the drawbacks of high cost, unreliability and non constant output, in addition, at the onset of this work, they were not available commercially. The five curies of PuBe sources tried did not provide sufficient neutron flux for good statistical accuracy. The availability of Cf^{252} (a synthetic transuranium isotope) makes the use of isotopic thermal neutron activation analysis a possibility.

During the summer of 1971 a laboratory shield activation assembly was designed and constructed. This shield assembly was designed to hold a 5 milligram Cf^{252} source which was obtained from the A. E. C. Savannah River Facility under the Market Evaluation Program. This system, designated the SST consisted of a three foot (O.D.) stainless steel

³F. A. Iddings, Arman, Et Al., "Nuclear Techniques for Cement Determination," Highway Research Record, No. 268, 1969, pp. 118-130.

tank with lead, water, boric acid, and concrete block as the primary radiation shielding materials (see Appendix 1 for further design information). The primary objective of this phase of the project was to determine the baseline parameters such as the required neutron flux for reliable analysis. The SST provided a variable geometry with respect to the moderator thickness and source-to-sample distance. In this assembly the source was raised from the bottom of the tank to an activate position determined by an elevator stop and lowered to end activation. The parameters determined in the SST were source size, amount of moderator between source and sample, and the activation, delay and counting time schedules. Another parameter determined was a comparison of detector [NaI(Tl) crystal] sizes.

The SST was provided with a remote operating elevator for moving the source into the activation position and an automatic release for ending activation. This was necessary to reduce the operator exposure rates measured near the assembly when the 5 milligram source was raised to a position between one and two inches from the surface of the water shield. The exposure rates measured over the shield assembly with the source in the activation position were in excess of 5000 mR/hr. and near the door to the room at waist level of 150 mR/hr. Through the use of the remote-automatic activation system and limitation of residence time the average operator exposure for a one month period was less than 100 mRem.

The results obtained from studies of soil cement activation analysis using the SST showed:

The response obtained from activating mixtures of 100% soil, 95% soil--5% cement, 90% soil--10% cement and 85% soil--15% cement formed a linear calibration curve. These values were reproducible.

This calibration curve allows analysis of an unknown sample through use of a calibration curve. (See Figure 3-1.)

The calibration curves obtained for different types of soils mixed with Portland cement were parallel. The differences in the counts observed for different soil types were due to differences in the natural calcium content of the soils (see Figure 3-2). The slope of the calibration curve was dependent on the neutron flux (the higher the flux the greater the slope of the curve). From the data observed a flux of 3×10^6 n/cm² - sec was determined to be the minimum flux desirable to obtain 1 - 2% (based on cement content) accuracy in field or laboratory cement content analysis. The results observed can be seen in Figure 3-3.

The size of the detector to be used was also studied. The detector size was varied (3 inches x 3 inches, 4 inches x 4 inches, and 5 inches x 5 inches) and samples were activated using constant geometry flux and timing schedules. The results of these experiments showed that the increase in response shown by the 4 inches x 4 inches over the 3 inches x 3 inches was small compared to the extra cost involved. The 5 inches x 5 inches, however, showed a greater relative increase than the 4 inches x 4 inches over the 3 inches x 3 inches (see Figure 3-3). From this data the 5 inches x 5 inches NaI(Tl) crystal detector was selected for use over the remainder of the research period. The greater sensitivity of the 5 inches x 5 inches crystal allowed a higher accuracy for a small cost compared to an increase in source size.

Based on the designed parameters determined using the SST and the shielding requirements discussed in Chapter Two, the design of the field-portable Activation Shield Assembly was optimized and the assembly was constructed (see Appendix 2 for construction details). After

Figure 3-1

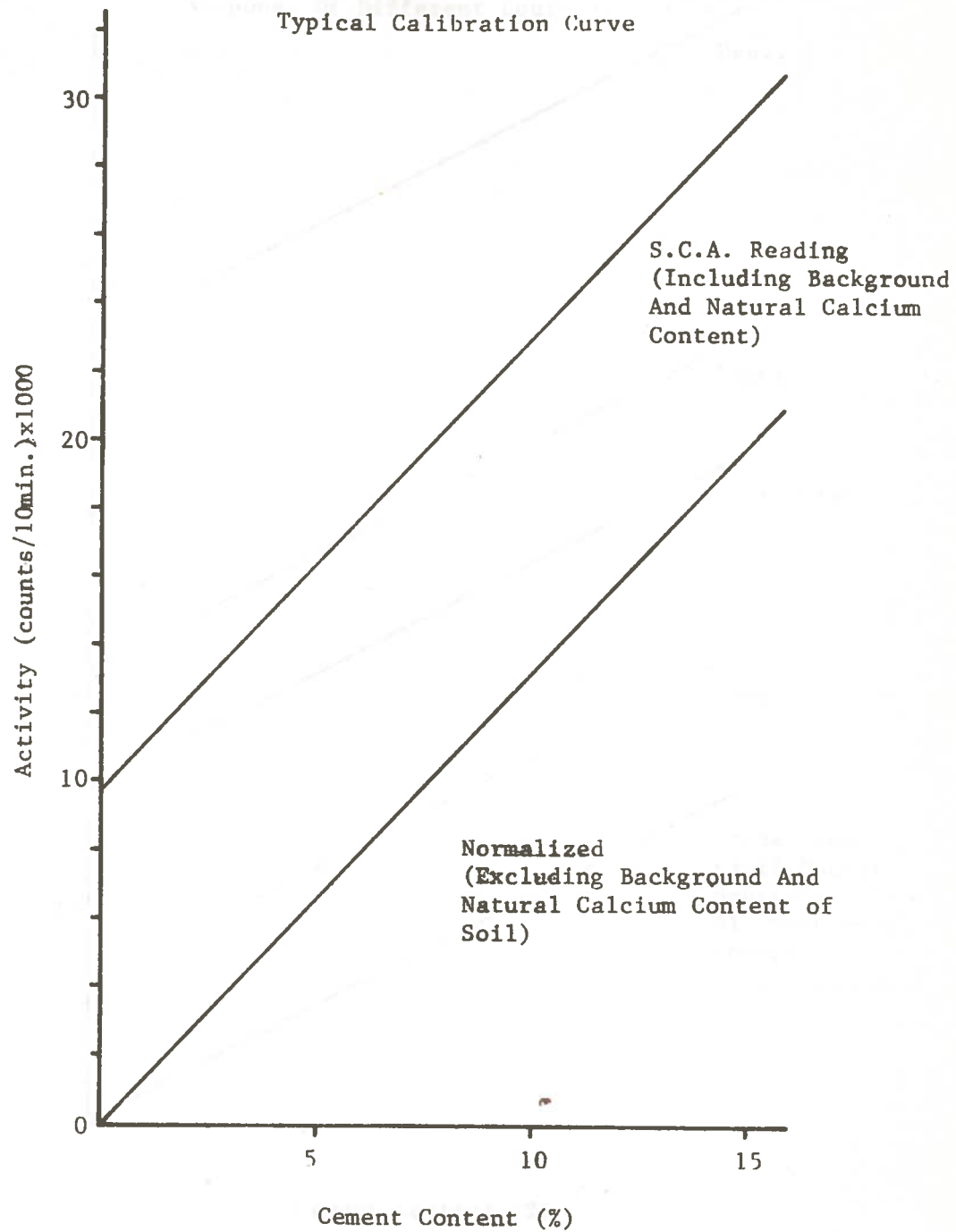


Figure 3-2

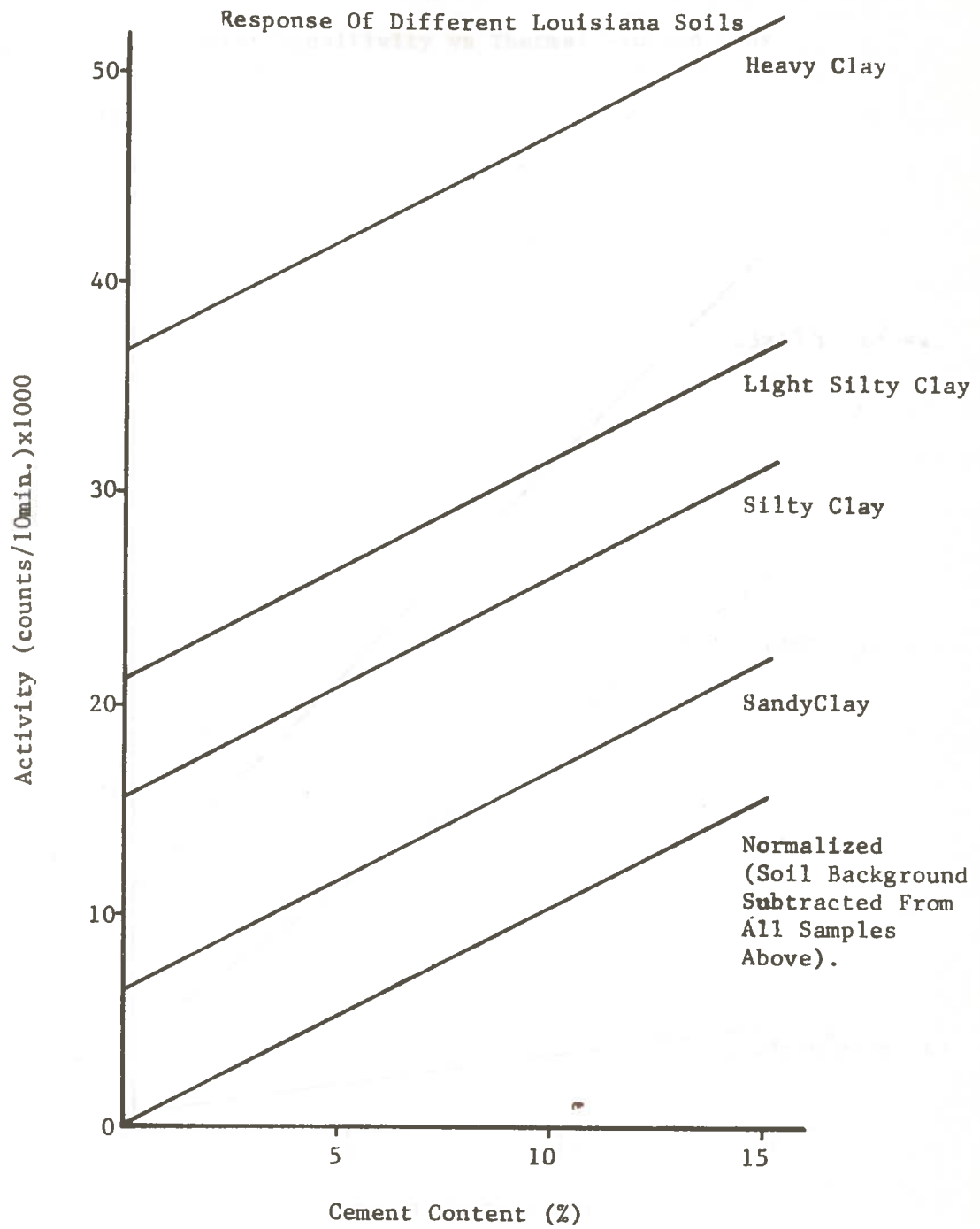


Figure 3-3

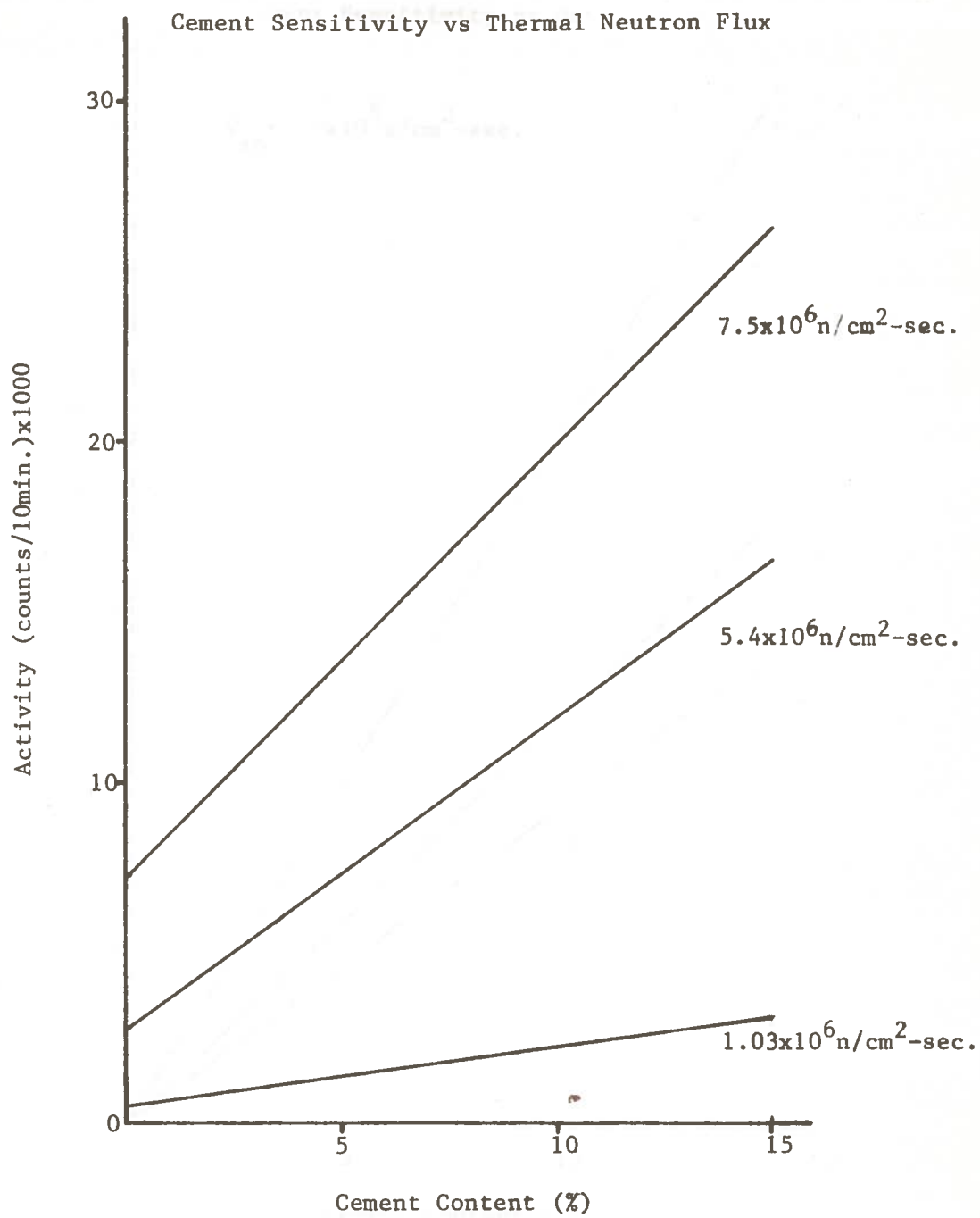
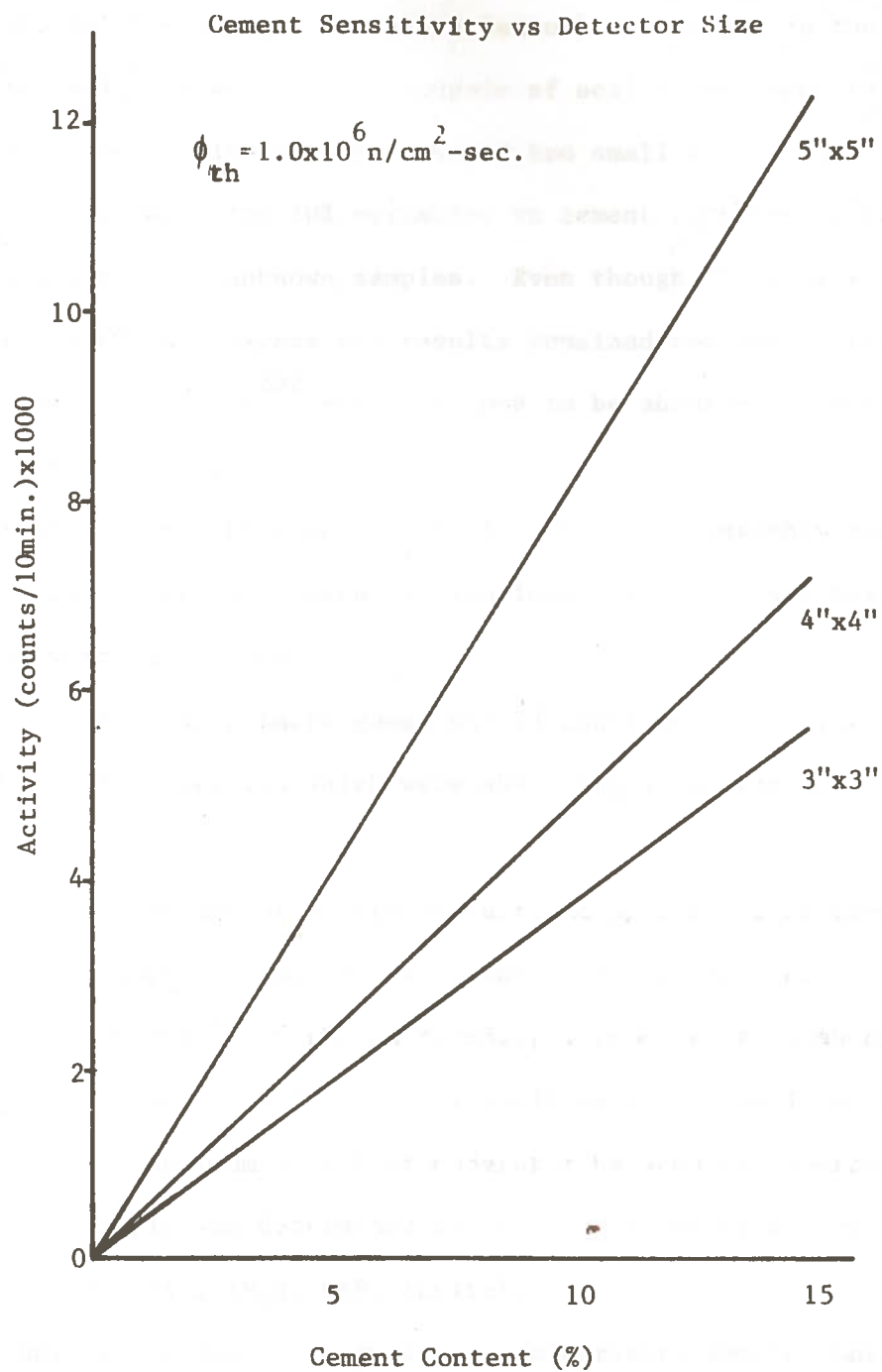


Figure 3-4



completion of construction a 200 microgram Cf²⁵² source (obtained from the L.S.U. Californium Demonstration Center) was loaded in the Activation Shield Assembly and activation analysis of soil cement samples was attempted. The results obtained showed too small a variation in response (2000 counts/10 min. for 10% variation in cement content) to provide accurate analysis of unknown samples. Even though the source size was increased to 600 micrograms the results remained too small; the neutron flux provided by the Cf²⁵² source seemed to be absorbed by the shield rather than by the sample.

At this point, this source shield activation assembly configuration was termed a failure and investigation into the causes was begun. The following were discovered:

1. The lead primary gamma shield contained impurities of high cross section which were absorbing a substantial number of neutrons.
2. The amount of moderator surrounding the source (hemisphere 5 inches diameter) was found to be insufficient.
3. The BWP^{4/} of the surrounding volume was also absorbing the neutron flux before they could be scattered into the sample.
4. The optimum amount of moderator between the source and the sample was determined to be 1.25 inches of homogeneous material (H₂O, WEP, lucite).

These observations led to a new source, moderator, sample configuration. (See Figure 3-5.) Two experiments were conducted to determine the usefulness of this arrangement. The first was a quick, simple experiment

⁴Borated WEP compound (3 mg. of boron as boric acid/cc.).

to set up and perform. It consisted of a source in the above configuration in a pail of water with a 29 microgram Cf^{252} source (see Figure 3-6). Data taken from activation of soil cement samples showed that this source arrangement would provide sufficient neutrons for activation analysis techniques.

As a second check a larger assembly was constructed. It consisted of a 55 gallon drum filled with WEP. The Cf^{252} source was positioned about 15 inches from the bottom and in the center (see Figure 3-7).

Again the results observed showed that this arrangement was useful. The results of activating samples in the spherical assembly and with the two experimental arrangements can be seen in Table 3-1.

Table 3-1

	100% soil	90% soil + 10% cement*
Activation Shield Assembly	0	1954
Bucket	0	8434
Barrel	0	17801
Activation Shield Assembly (final)	0	14069

*10 min. counts after 10 min. irradiation and a 1 min. delay and normalized to zero for background and Ca content of soil.

The large changes in the differences between the count observed for this 100% soil and the 90% soil, 10% cement samples increase greatly. This means that the slope of the calibration curve is increasing, resulting in a higher accuracy for the determination of cement content.

After consideration of this information it was decided to modify the first spherical assembly to conform to this source arrangement. The new arrangement required the removal of the Pb shell and removal of the BWEP within 7 inches of the source. (For details on new configuration,

Figure 3-5
Source-Lead-WEP Assembly

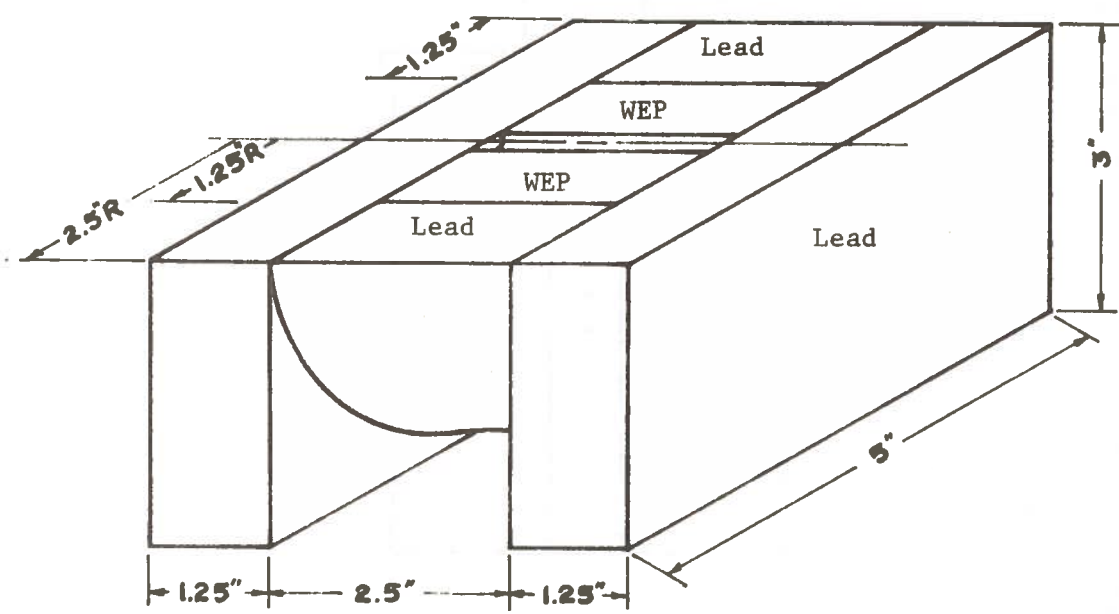


Figure 3-6
Bucket Irradiation Assembly

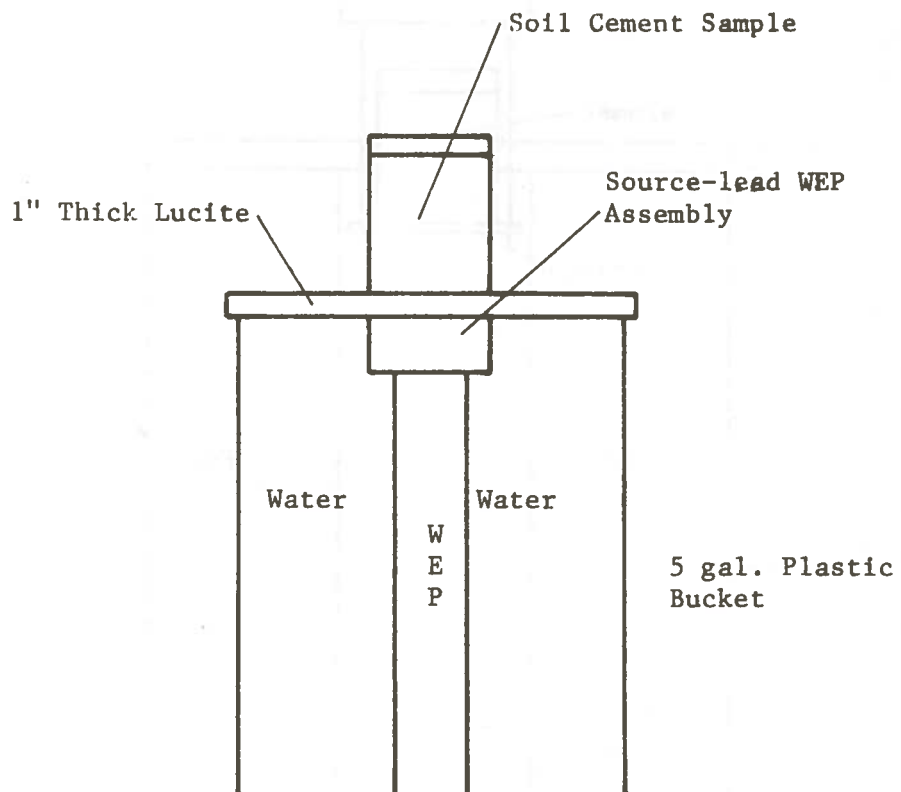
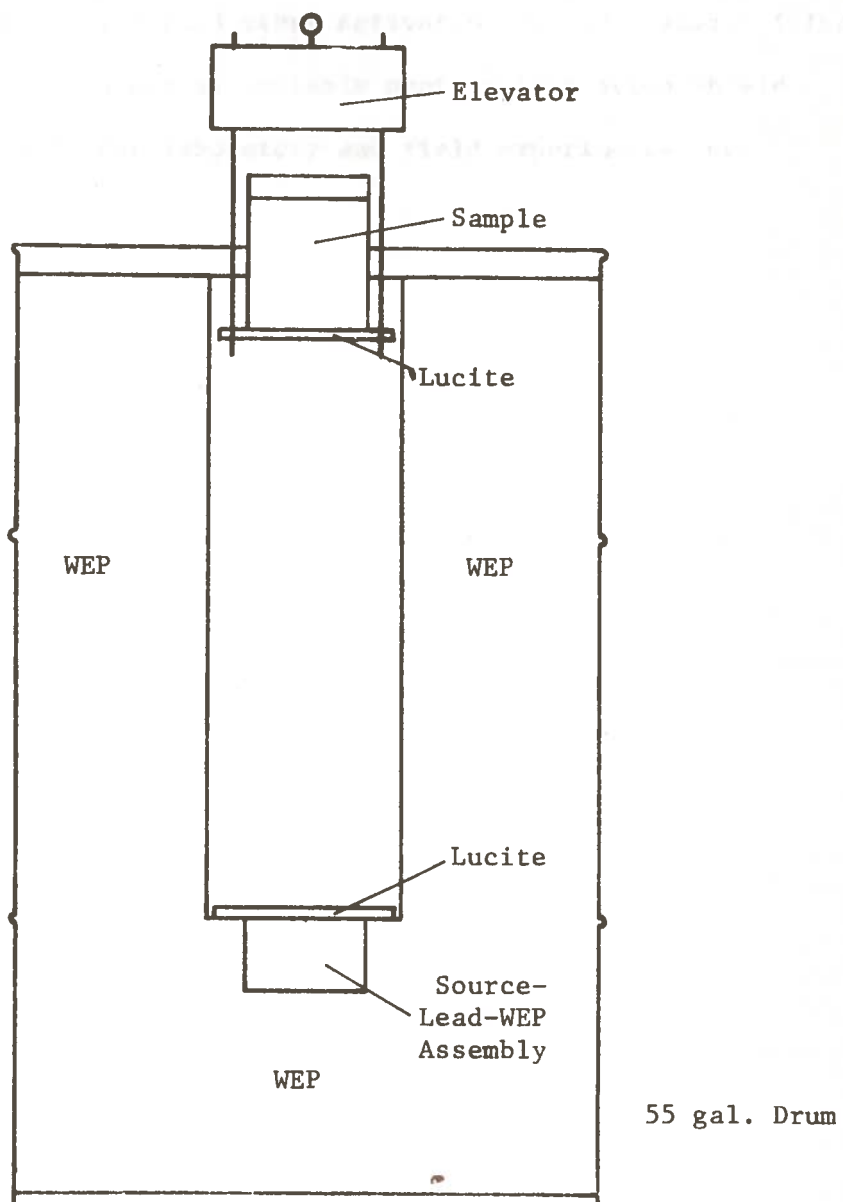


Figure 3-7
Barrel Assembly



see Appendix 4.)

The results obtained after modification of the spherical assembly can be seen in Table 3-1 listed under Activation Shield Assembly (final). These data showed that a useful portable neutron Activation Shield Assembly was available for laboratory and field experimentation.

CHAPTER FOUR

Results

For a period of just over 12 months, the portable soil cement spherical activation shield has operated dependably in the field providing reproducible geometry and predictable neutron flux. Over 760 samples have been irradiated in this assembly during the past year. While its primary use has been to activate soil cement samples, it has also been used to provide short lived isotopes for classroom and student laboratory studies. It has also provided activation neutrons for preliminary studies on concrete. These laboratory activation analyses of about 40 concrete samples provided information used to obtain an extension of the research contract. The extension will be used to study wet mix concrete sampling and analysis techniques.

The only changes that have been made in its original design consist of the changes in the moderator absorber geometry to provide sufficient neutron flux for soil cement analysis (design change discussed in Chapter Three) and a new cart assembly. The old cart assembly proved too wide for most enclosed trailers readily available. The new cart assembly consists of a wooden frame with casters to provide easier handling.

The performance of the activation shield assembly in either the field or laboratory environment has been without major down time incident. The only problem that occurred was a loss of the sample shelf in the carousel due to failure of two 1/8 inch diameter plastic pins. The weight of the storage plug was too much for the original shelf. These pins were replaced with 1/8 inch diameter steel screws

in the laboratory (where most of the experimental work was performed). The Activation Shield Assembly storage area was further shielded by concrete blocks to reduce the exposure levels to personnel working in the engineering laboratory area of the Nuclear Science Center.

Laboratory data obtained from activation of soil and soil cement samples yielded a linear relationship between counts/time and cement content (see Figure 4-1).

Laboratory studies (activation analysis of over 630 samples) showed that the depth of penetration (thickness of sample from which a useable return of activity can be measured) was in excess of 5 inches (see Figure 4-2). This information reinforced the use of the 1/2 gallon paper ice cream cartons (6.5 inches tall) as sample containers.

The results of laboratory studies also showed that: the calibration curves for different soil types were linear, parallel and could be superimposed (see Figure 4-3), the average error (in cement determination) was $\pm 5\%$ of the cement added to soil cement samples, operator training required on the average one eight hour day to complete, and the largest source of error (which occurred on about 5% of the samples analyzed) was operator timing.

Experience gathered on field trips showed that the major problem encountered was the difficulty in moving the 800 pound Activation Shield Assembly into and out of an enclosed 4 foot x 6 foot trailer. In the field environment, as in the laboratory, the single major source of error was in timing, either timing the activation or the delay time before counting.

More than 100 samples were analyzed during four field trips. The data collected showed, again, that the Activation Shield Assembly

Figure 4-1

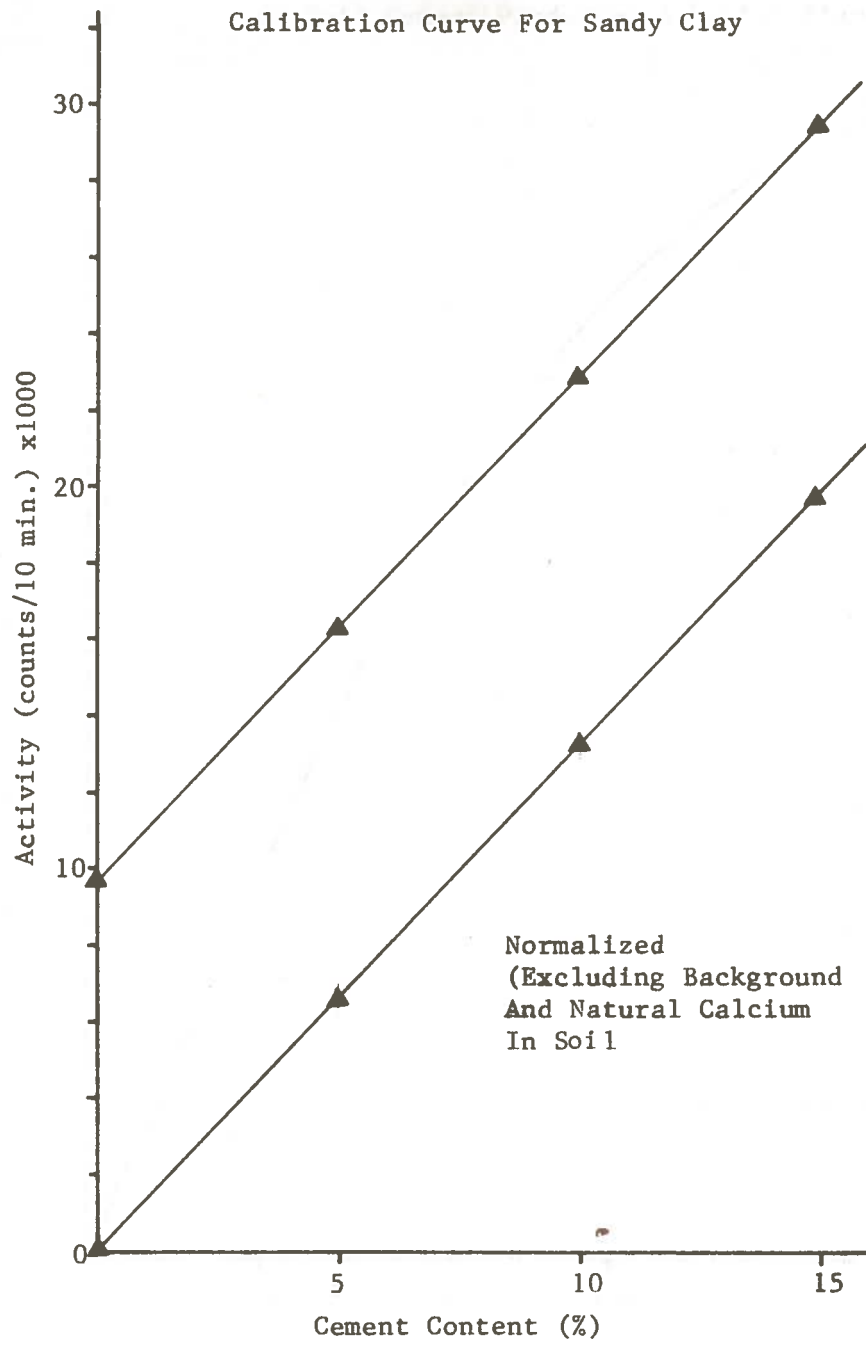


Figure 4-2

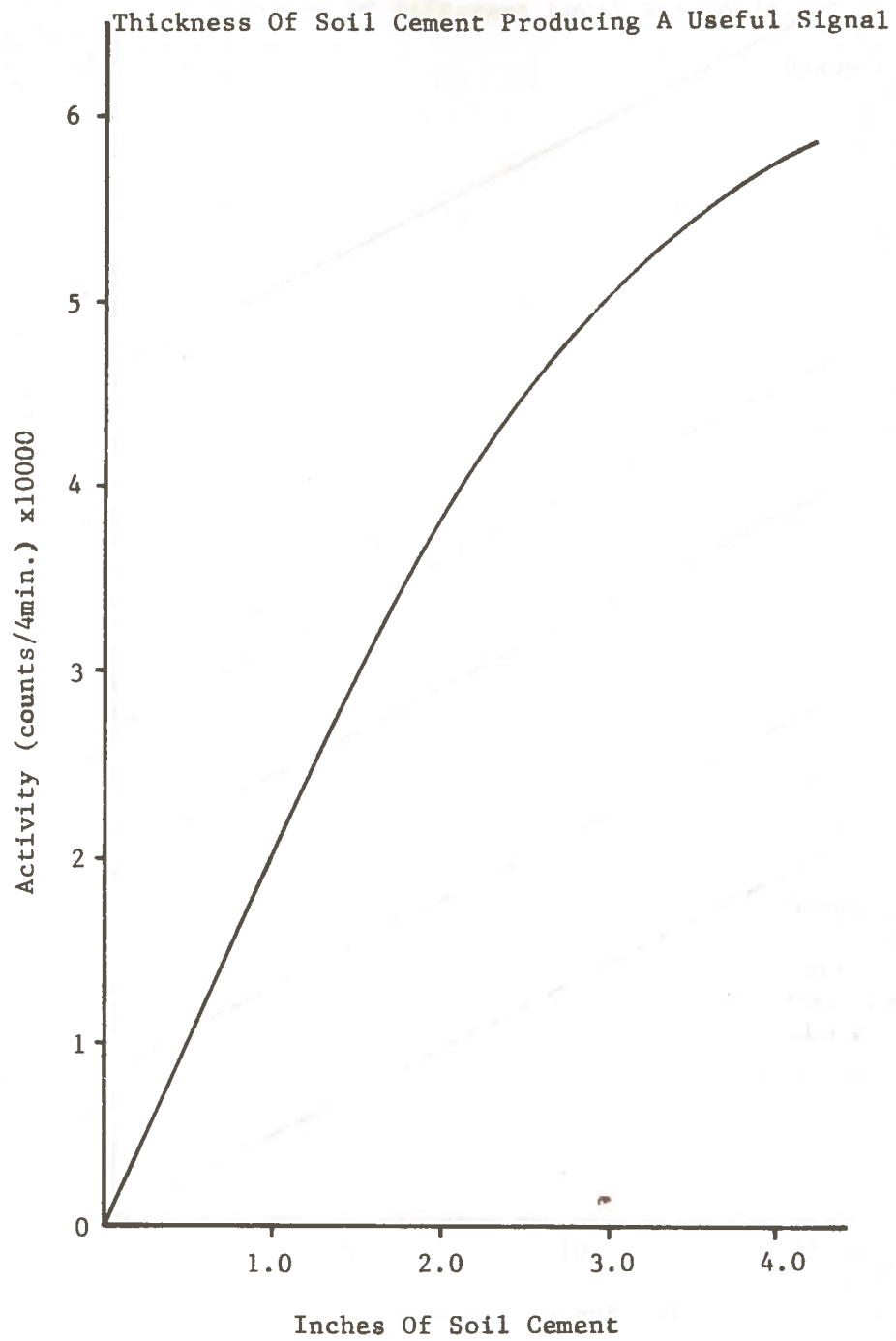
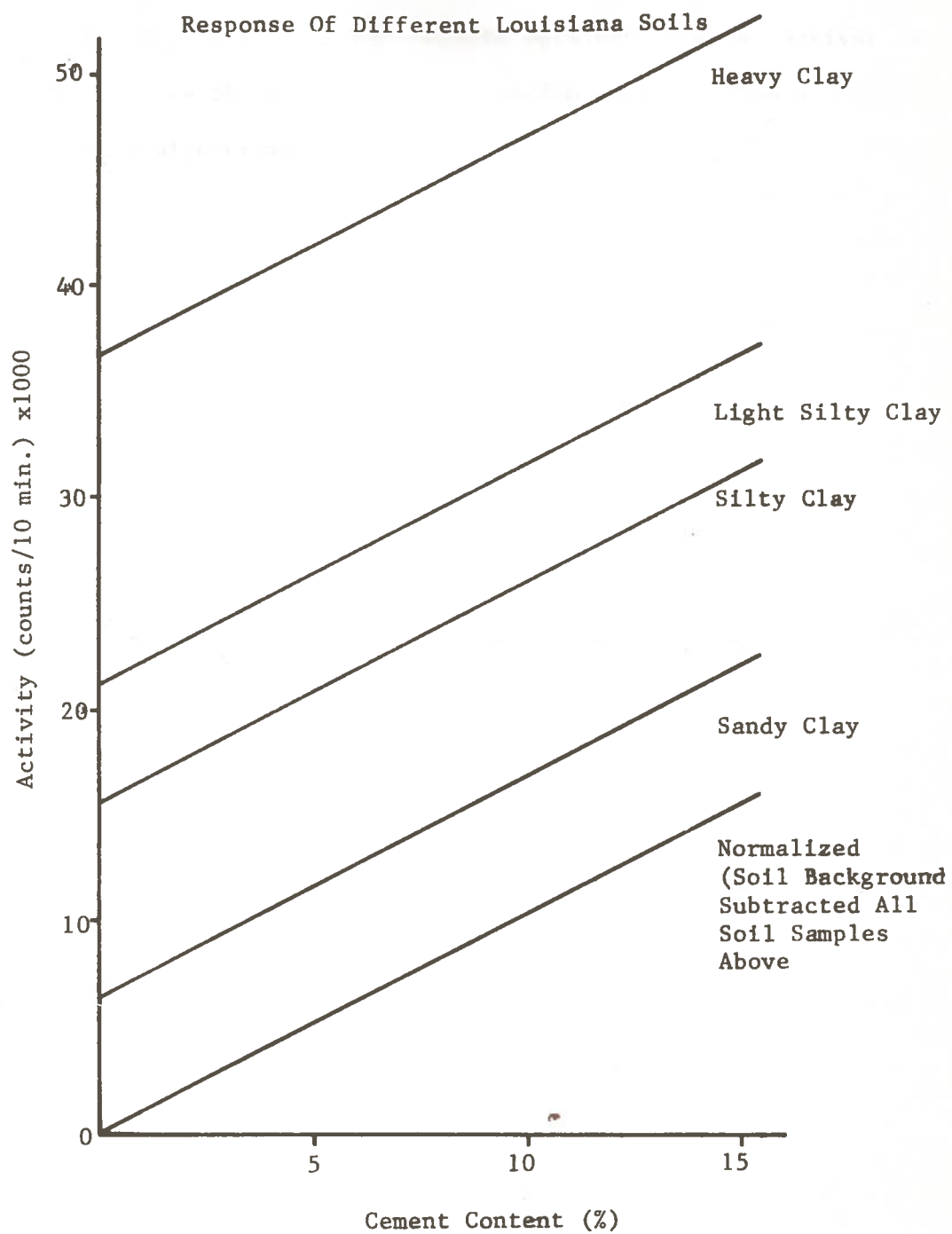


Figure 4-3



provided a constant flux, constant geometry activation environment for the soil cement samples. The results obtained from the activation of over 760 samples showed that the activation shield assembly satisfied the design criteria.

CHAPTER FIVE

Conclusions

Five conclusions can be made from the results obtained from the design, construction, and operational experience over a period of two years.

A representative soil cement sample should be of a two to three kilogram mass (5 inches diameter x 6.5 inches tall). This sample size minimizes the effects of inhomogenities such as soil or cement lumps, large gravel, or organic material included in the sampling.

A Californium²⁵² source of between 100 and 200 micrograms can provide sufficient neutron flux for activation analysis of representative soil and soil cement samples.

A spherical shield of 32 inches diameter weighing about 700 pounds and consisting of WEP and BWEP (borated WEP) provides sufficient biological shielding for operators.

Cement content analysis of soil cement samples can be provided to a precision of $\pm 5\%$ of the cement added to the sample. This provides a reasonable precision within the present practices of highway construction ($\pm 2\%$ cement). The cement content analysis precision is much greater than that which can be obtained by present field mixing techniques (mixing soil, water, and cement on or at the construction site).

A concise simple cement analysis can be performed by relatively untrained operators in a field environment in 10 to 20 minutes per sample. The possible cross calibration of soil types would even further simplify the analysis technique. This can be accomplished

since the normalized calibration curves for different soil types superimpose on one another.

The following recommendations are made based on field and laboratory experience obtained while working on this project.

The timing of the activation of samples should be automated. This would reduce the possibility of operator error.

The shield activation assembly should be permanently mounted in a sole-use enclosed truck or trailer that can be locked and stored in a restricted area. This would reduce the physical hazard to operators loading and unloading the assembly into a trailer.

The fiber glass outer container should be replaced on future assemblies by a painted carbon steel shell to reduce the possibilities of damage while in transit. This could also provide partial compliance with Department of Transportation regulations concerning transportation of hazardous radioactive materials.

Further studies should be made to extend this type of analysis to include concrete (plastic or cured in place) with the possibility of a single activation shield assembly and counting system for both.

Further studies should also be made to determine the effects of varying sample diameter. This would allow the cross calibration of sample cores currently being taken for destructive testing.

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Texas.

APPENDIX 1

Construction Of SST Assembly

The following steps will result in the construction of a laboratory activation assembly.

1. Fabricate the lucite inner tank assembly as shown in Figure A1-1.
2. Fabricate the sample holder and source stop assembly as shown in Figure A1-2.
3. Fabricate the tank lid as shown in Figure A1-3.
4. Fabricate the elevator assembly as shown in Figure A1-4.
5. Fabricate the tripod as shown in Figure A1-5.
6. Assemble the sub-assemblies as shown in Figure A1-6.
7. The automatic release timer is constructed as shown in Figure A1-6.

Figure A1-1

Inner Tank Assembly

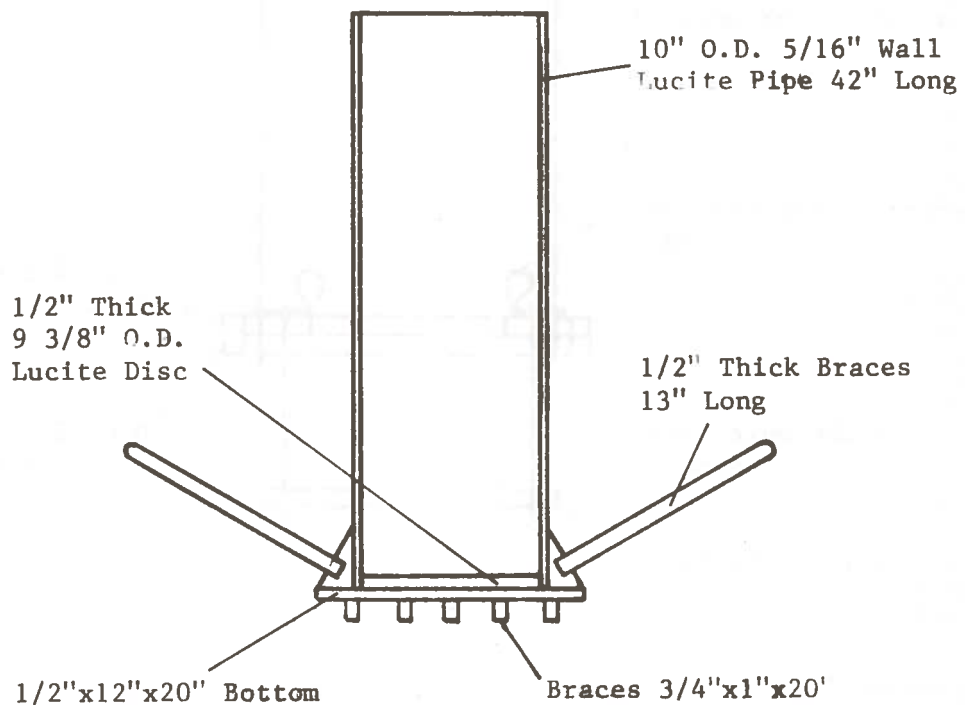


Figure A1-2

Sample Holder And Source Stop Assembly

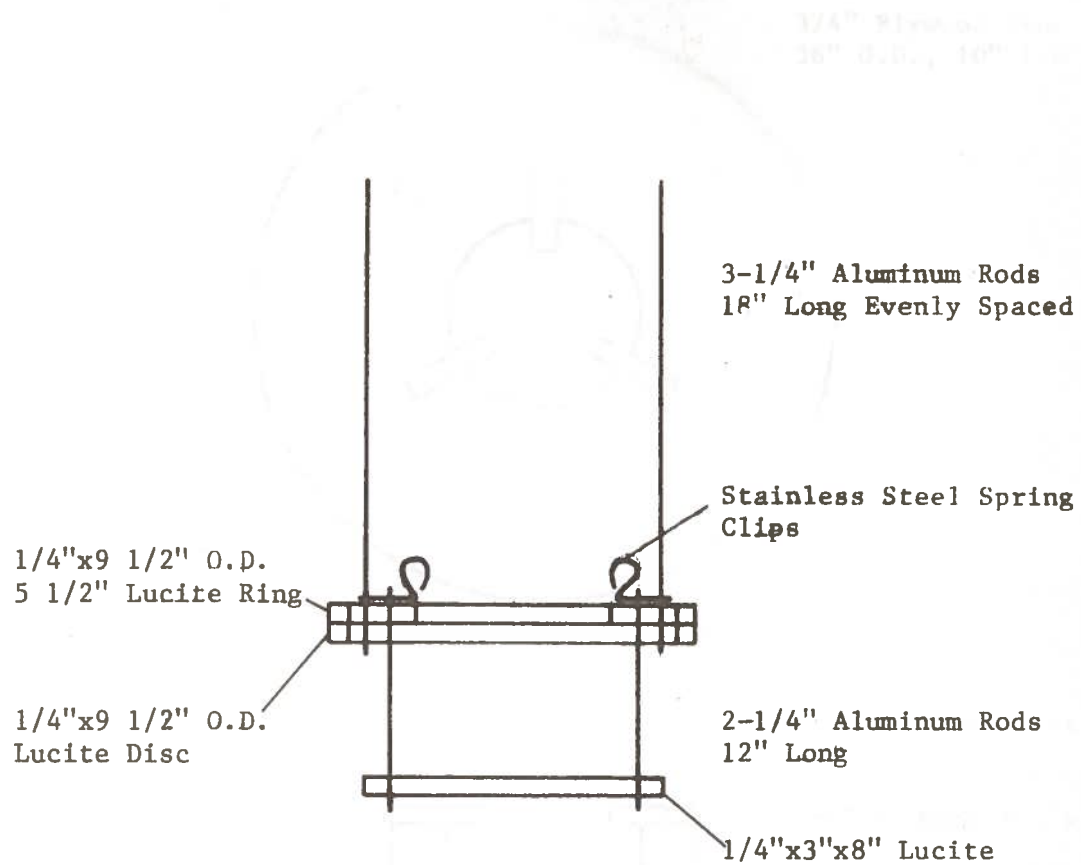
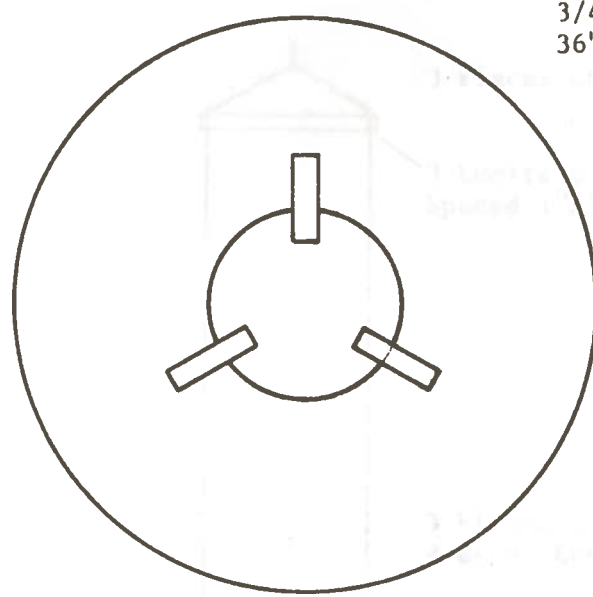


Figure A1-3
Tank Lid Assembly



3/4" Plywood Ring
36" O.D., 10" I.D.

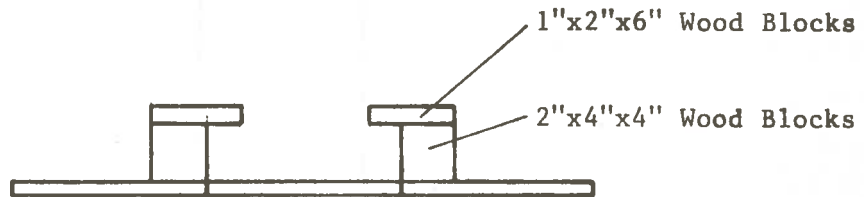


Figure A1-4
Elevator Assembly

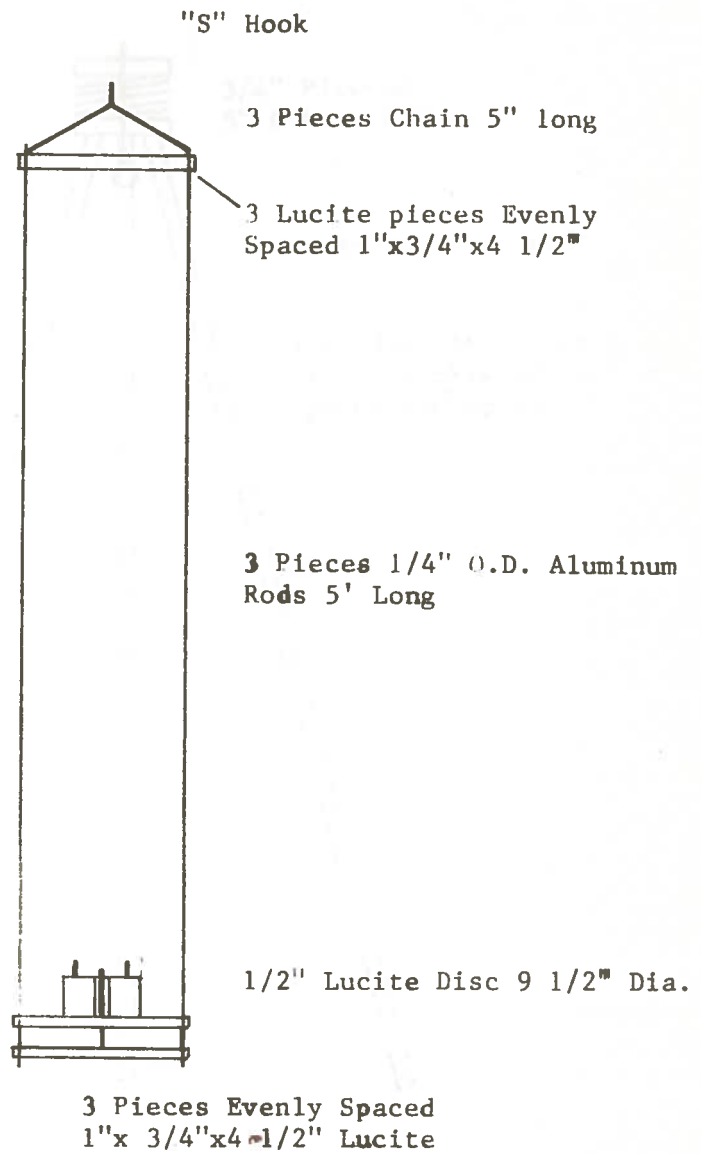


Figure A1-5
Tripod Assembly

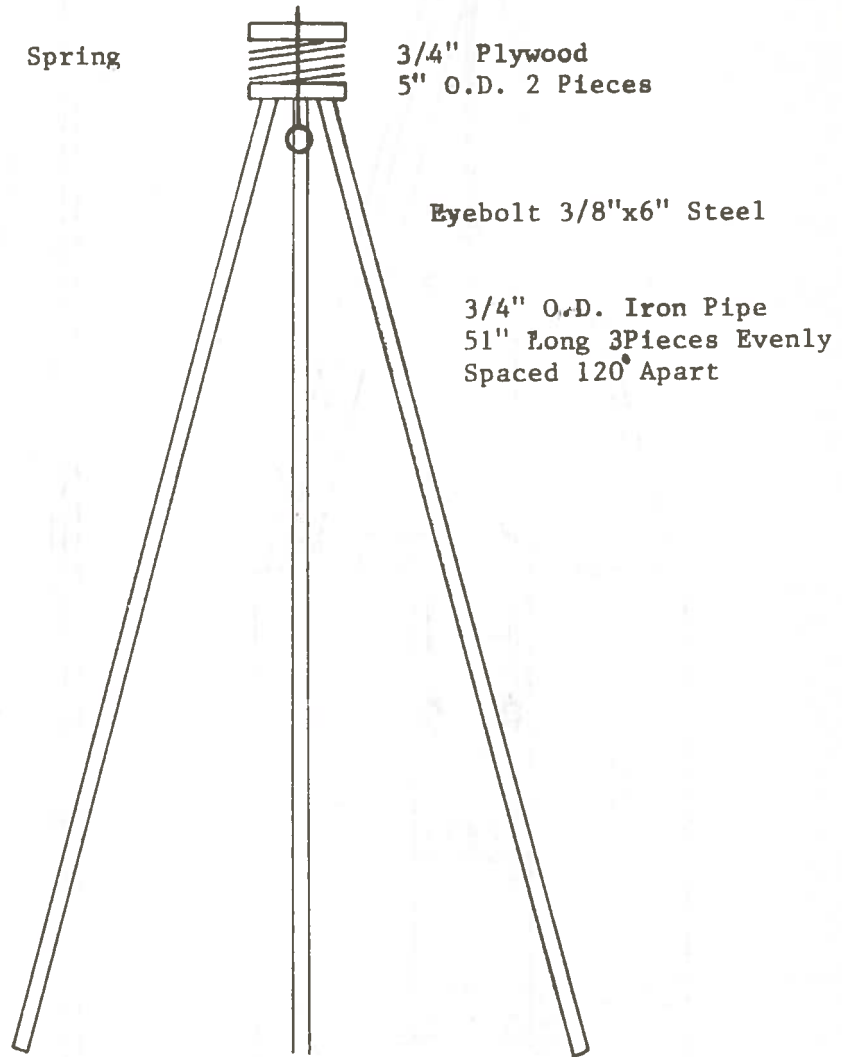
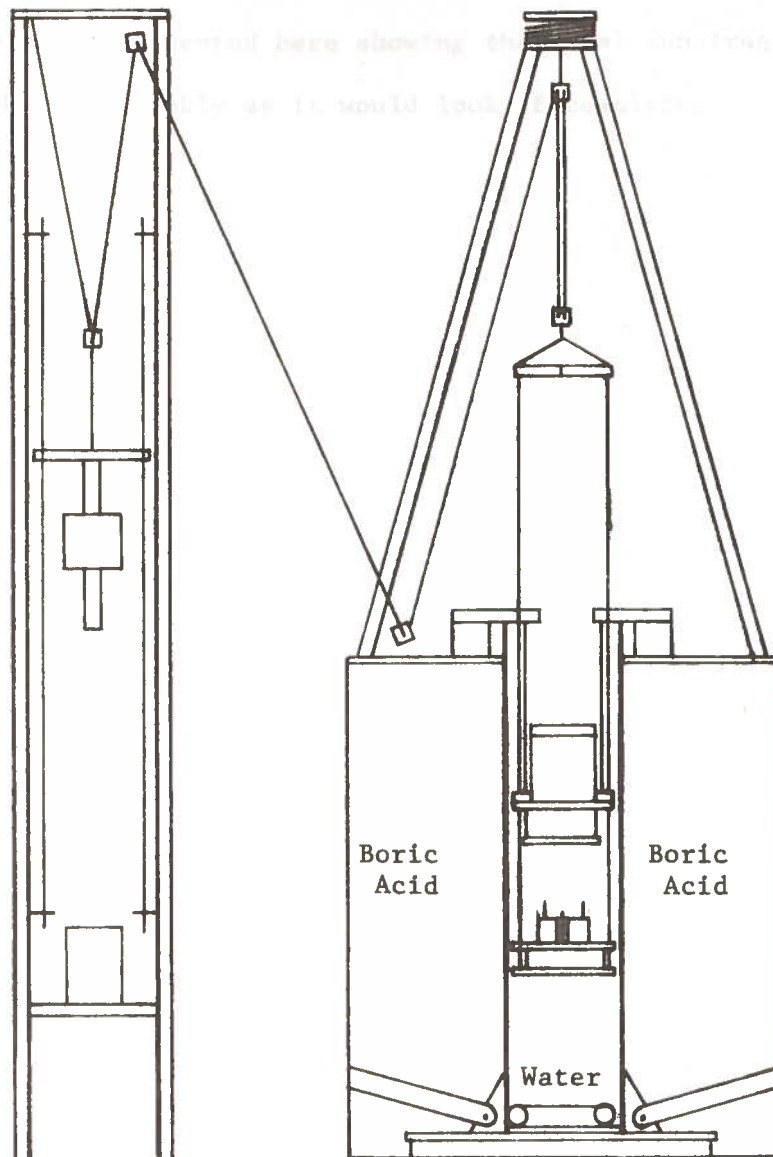


Figure A1-6

Stainless Steel Tank Irradiation Assembly



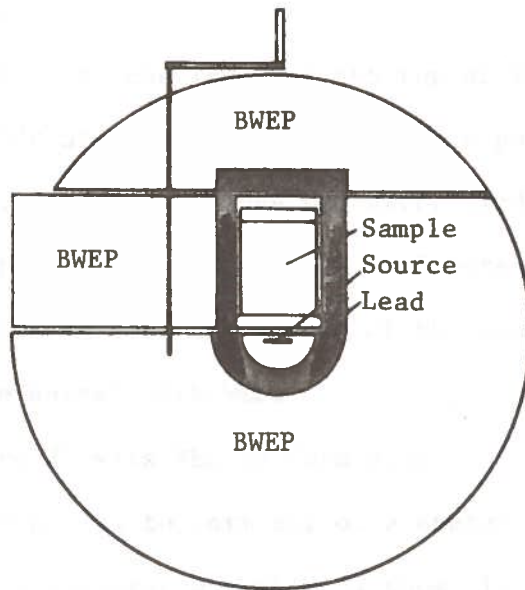
APPENDIX 2

Construction of Activation Shield Assembly

A drawing is presented here showing the final construction of the Activation Shield Assembly as it would look if completed.



Figure A2-1
Activation Shield Assembly



APPENDIX 3

Construction Of The Barrel

The following steps are necessary for construction of the barrel activation assembly.

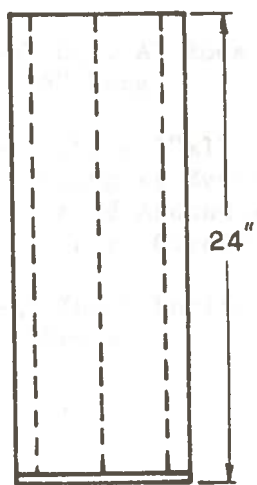
1. Fill 1/3 of a 55 gallon drum with WEP.
2. Place the lead shield assembly on the center of the WEP surface.
3. Pour WEP up to the level of the top of the lead shield.
4. With a 3/8 aluminum rod in the source position pour the cup full of WEP. (Remove rod after hardening)
5. Fabricate the two forms shown in Figure A3-1.
6. Place form #2 over the center of the source position and fill the barrel with WEP.
7. Fill form #1 with WEP to form plug.
8. Cut 3 inch plug to form top of elevator assembly.
9. Construct elevator assembly as shown in Figure A3-2.

Figure A3-1
Triangular WEP Forms 1 & 2

Aluminum Foil
on Outside



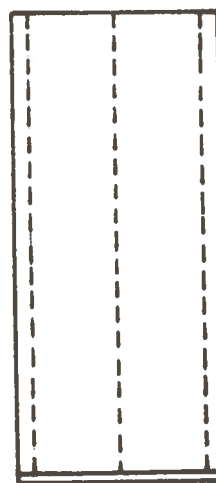
Aluminum Foil
on Inside



Form 1



3/4" Plywood
Bottom



Form 2

Figure A3-
Elevator Assembly

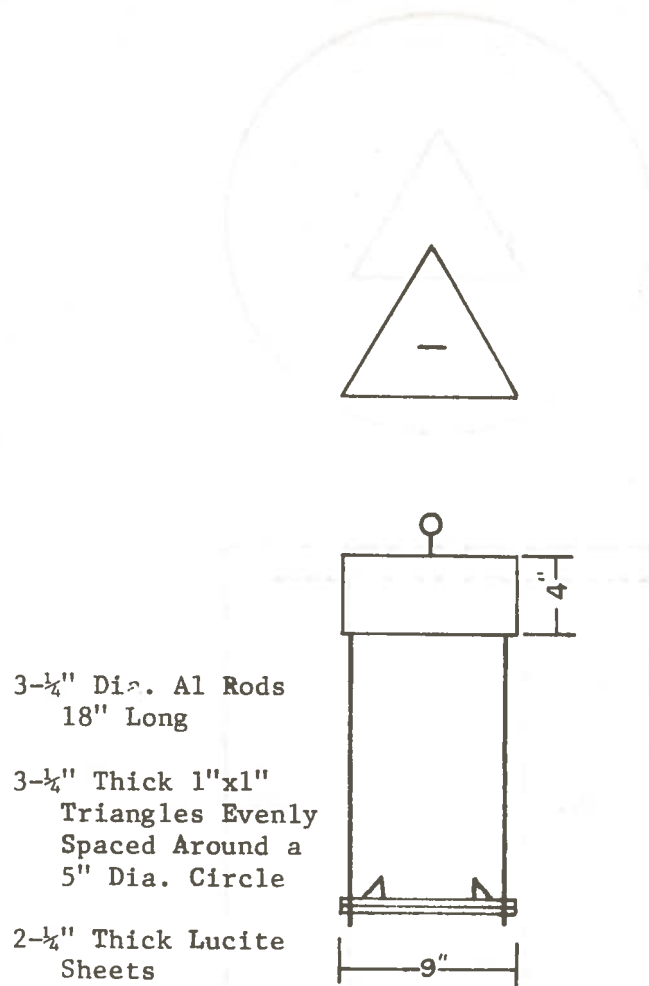
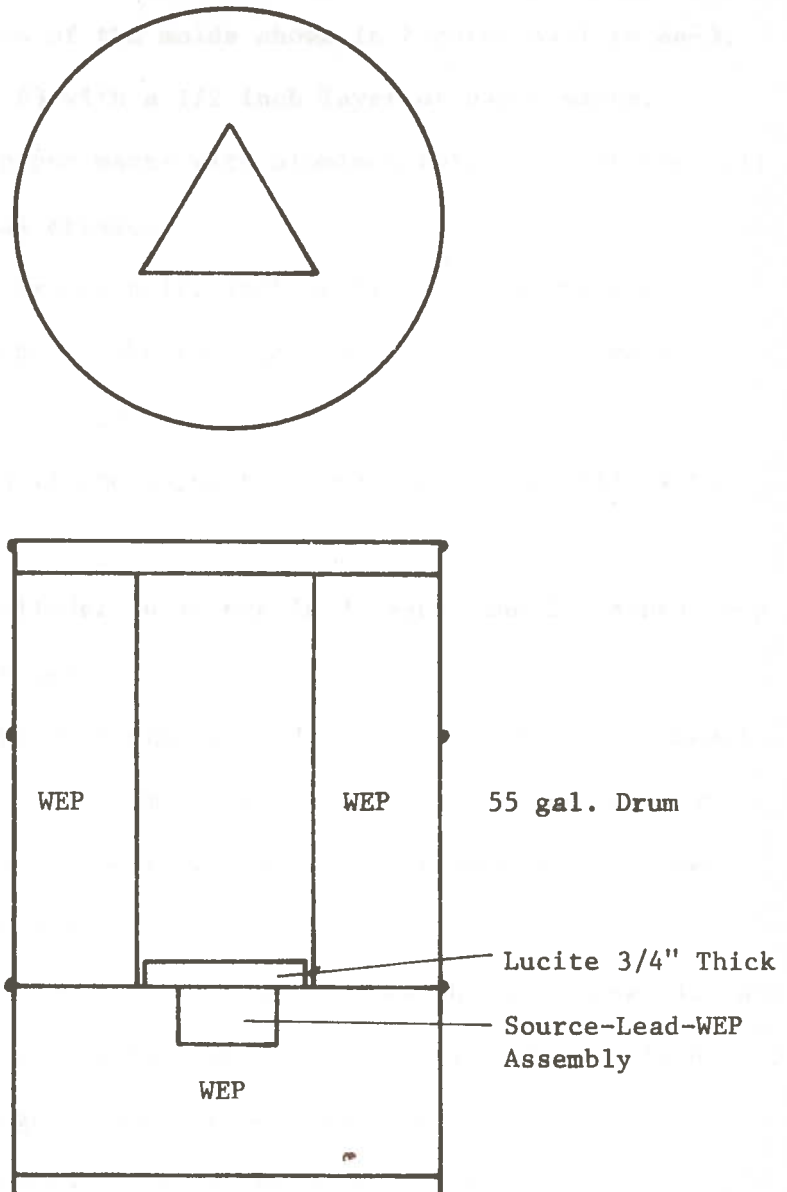


Figure A3-3
Barrel Assembly



APPENDIX 4

12. Construction Of The Spherical Activation Shield Assembly

After construction of the molds shown in Figures A4-1 to A4-3,

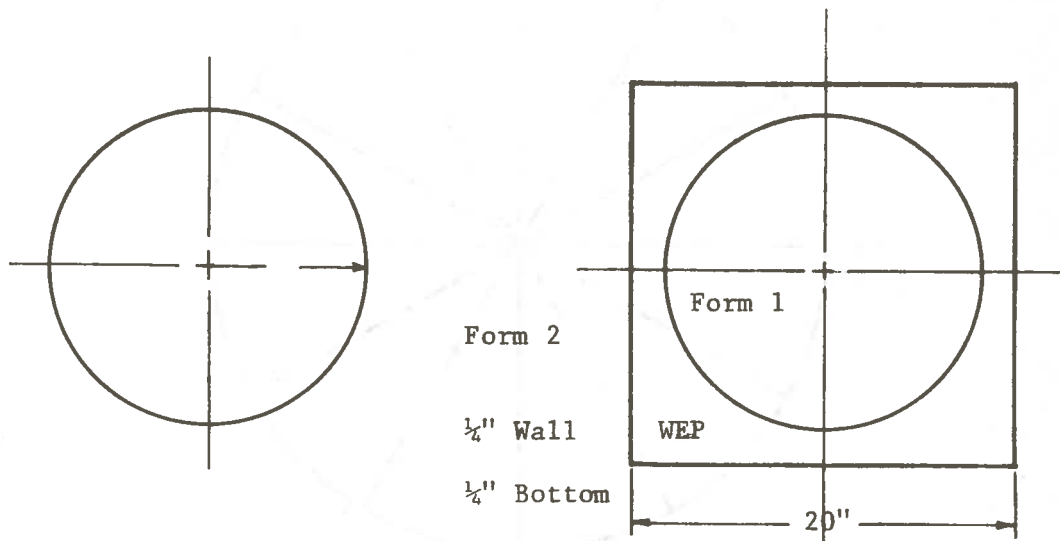
1. Cover mold #3 with a 1/2 inch layer of paper mache.
2. Cover the paper mache with aluminum foil and coat the foil with silicon grease.
3. Cover the form with 1/2 inch of fiber glass resin and cloth (building the thickness with 1/8 inch layers). Make two fiber glass shields.
4. Take one shell and support it open-end up and fill with BWEP.
5. Remove a cylinder 14 inches in diameter and 10 inches deep from the center.
6. Position the Pb source shield in the center and the bearing race 5.5 inches from the source and fill volume with WEP.
7. Cut the remaining fiber glass shell transversely 8 inches from the open edge.
8. Fiber glass the ring like section to the bottom section and cut the ring to allow form #1 to fit with the 5/8 inch shaft through it and the bearing race.
9. Seal the cracks and fill the void with WEP.
10. Remove form #1 and replace the foil covering if damaged and fit it into the center of mold #2. Fill the void with WEP making mold number 4 (see Figure A4-4.).
11. Place the axle (5/8 inch steel rod with 1/4 inch aluminum re-bars) in mold #4 maintaining a normal position and position

- mold #5 against the side of the form, and fill with WEP.
12. Fill the fiber glass cap with WEP to complete pourings.
 13. Assemble as shown in Figure A4-5.
 14. Place the completed assembly on the cart assembly and secure.



Figure A4-1

WEP Molds
(Carrousel)



Form 1

Form 1 Inside
Form 2

1/4" Plywood Discs

Cardboard Covering

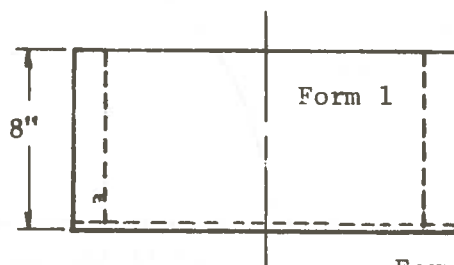
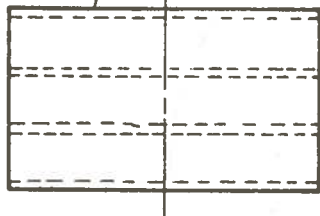


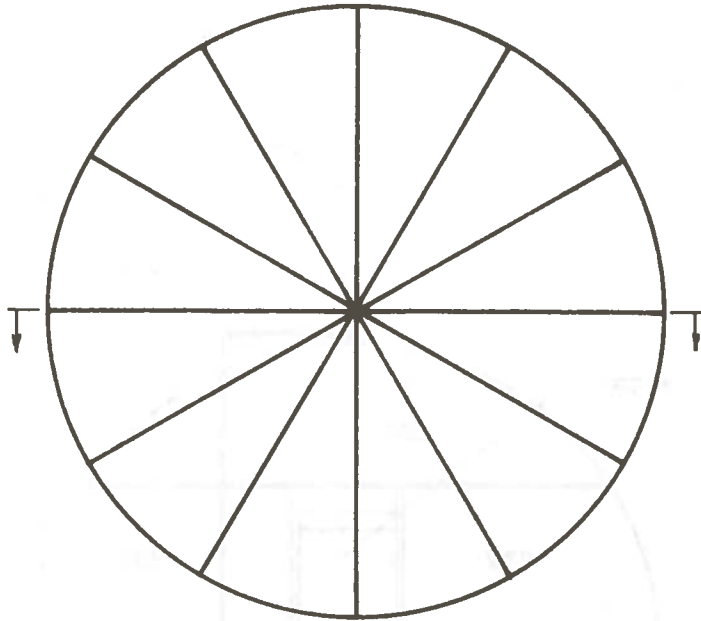
Figure A4-1a

Form 2

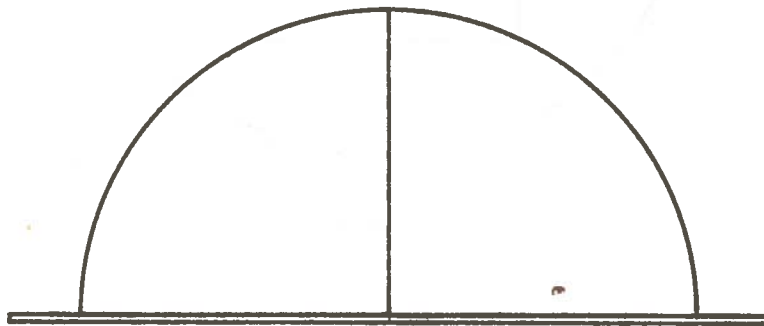
Figure A4-1b

Figure A4-2

Fiberglass Form



12 Plywood Sheets
 $\frac{1}{4}$ " Thick
16" Radius



Cover With Paper Mache and Al Foil

Figure A4-3
Modified Activation Shield Assembly

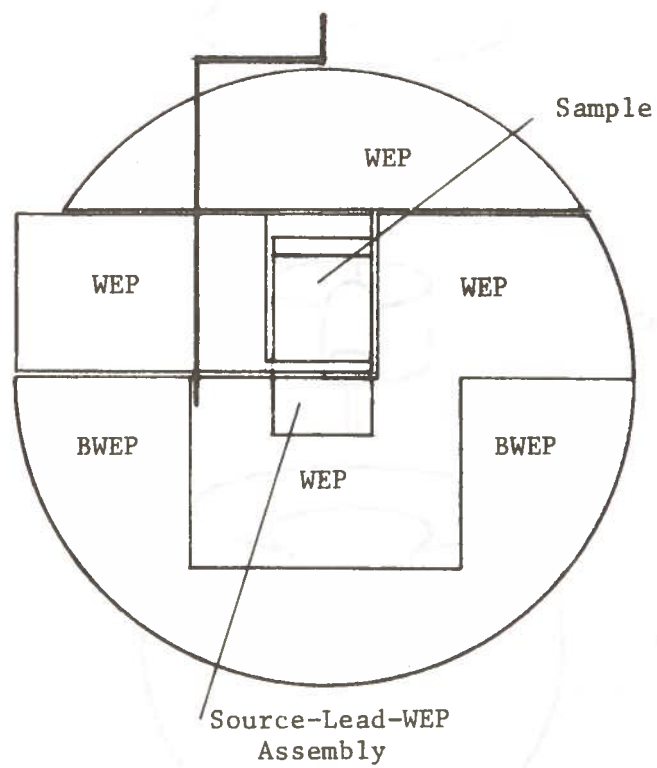
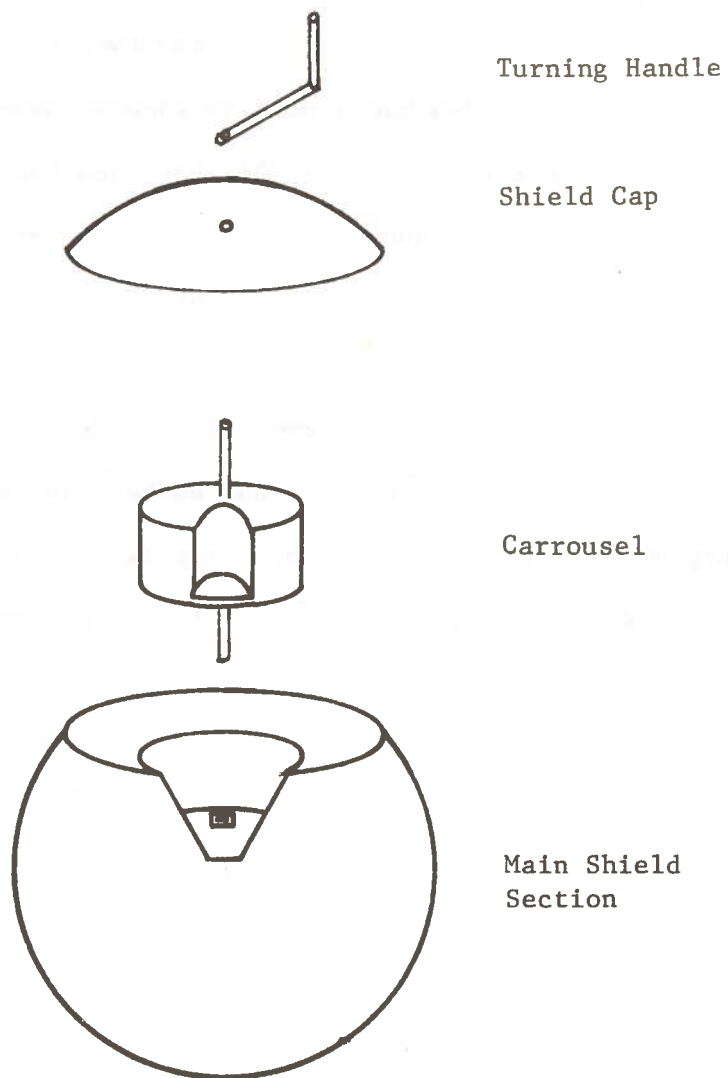


Figure A4-4

Exploded View Of Modified Activation Shield Assembly



APPENDIX 5

Sample Preparation

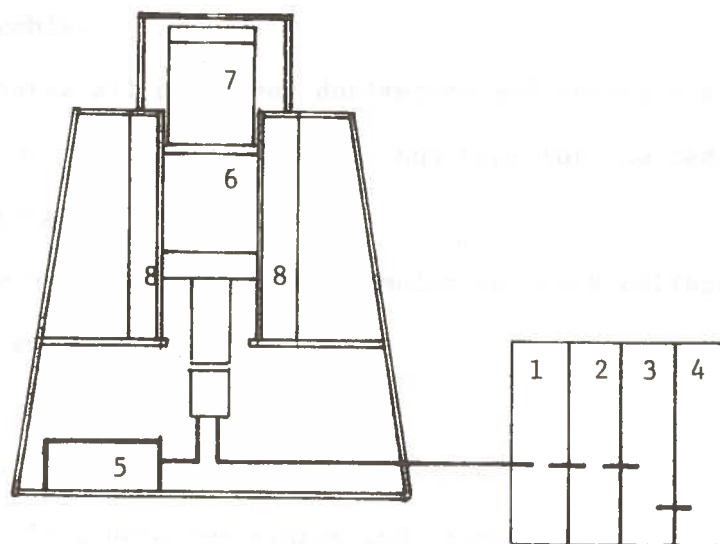
Laboratory samples were prepared as follows:

1. Soil dried for 24 hours at 200° F.
2. Soil crushed and powdered.
3. Mixed with known amounts of cement and water.
4. Mixtures weighed out into 2500 gram samples and packed into 1/2 gallon paper ice cream cartons.
5. Cartons sealed and labeled.

Field samples were prepared as follows:

1. Cartons filled at roadbed and labeled.
2. Samples weighed and weights reduced to 2500 gram \pm 100 grams.
3. Cartons sealed, and labeled as to origin, location and weight.

Figure A6-1
Counting System Diagram



1. Pre Amp-Amp-Discriminator
2. Single Channel Analyzer
3. Scaler
4. Timer
5. High Voltage Power Supply
6. Detector (5x5) NaI
7. Sample
8. Lead and WEP Shielding

APPENDIX 7

Operational Procedure

Before Going Into Field

1. Check the charge on all batteries.
2. Check the operation of equipment by running a test sample.
3. Measure the radiation levels around the Shield Activation Assembly.
4. Recharge all personnel dosimeters and record the reading.
5. Inventory the signs, locks, and rope for the radiation barricade.
6. Make up and run standard samples to check calibration of the system.

Field Set Up

1. Locate the activation/shield assembly at least 50 feet away from detector system and erect barricades with appropriate radiation warning signs.
2. Set up the detector and counting system and turn it on.
3. Check the single-channel analyzer (SCA) settings. Place a small Co^{60} source (5 μCi) microcurie on the detector. With the SCA in the "window" mode and the timer set for 10 seconds take counts with the upper level/window set at 1.0 and vary the lower level discriminator (LLD) until the 2.50 MeV sum peak is located. The LLD setting should then be reduced until the count rate drops to a minimum. If the LLD is further reduced, the count rate will increase. Set the LLD

where the count rate is at a minimum.

4. Now set the SCA to the "normal" mode keeping the LLD setting determined in 3. Set the upper level discriminator (ULD) to about 8.0. These LLD and ULD settings allow the measurement of gamma between about 2.4 and 8 MeV. For field measurements, reset the timer for a sample counting period of 4 minutes.
5. Activate and count standards made from the soil and cement in use. As a minimum, analyze the soil and two soil-cement percentages that bracket the composition specified. Activate samples from 4 to 8 minutes, allow them to decay 1 to 2 minutes, and count them for 4 minutes.*
6. Prepare a calibration curve from these data.
7. Begin activation/decay/counting procedures for the samples taken from the field. They should have the same weight as the samples in the standard set. Use the same activation, decay, and count schedule that was used for the standards.
8. Record field data including: sample origin, sample weight, counts per four minutes, and all instrument settings.
9. Compare the counts per four minutes to the calibration data found in step 6. From this determine the percent cement content in the sample.

Securing the Assembly

1. Lock the activation/shield assembly with a plug or old

*These time periods are set by sample type and cement specifications. Low sand and high cement samples may have activation times as short as four minutes. Samples with a high sand content should be allowed to decay for two minutes.

sample in the activate position when the assembly is not in use.

2. Read and record any radiation exposure to personnel from the dosimeters.

APPENDIX 8

Preparation Of WEP

1. Measure equal parts water and Alpon-661-P resin.
2. Measure sufficient hydrogen peroxide (30% concentration) to equal 0.75 to 1.0 percent of the total volume (water and resin).
3. Cool the water to about 70° F.
4. Pour the resin in a large mixing container and stir at a minimum of 1500 shear R.P.M.
5. Add a small portion of the water and mix until an emulsion is reached (the purple resin will start to turn a whitish color).
6. Continue adding the water in small portions, each time mixing until the color change is complete and even.
7. When the total mixture is a creamy white, add the hydrogen peroxide while stirring the mixture.
8. Mix until the mixture turns an even light green and pour into a mold (one to two minutes before hardening).

APPENDIX 9

Shielding Calculations

The neutron dose rate for Cf-252 can be expressed by:

$$D_n E (\text{mRem/hr}) = 23700M/r^2$$

where

M = micrograms of californium-252

r = distance in air from source

and the gamma (primary) dose rate can be expressed by:

$$D E (\text{mRem/hr}) = 1420M/r^2$$

Assuming WEP to have the same characteristics as water, the number of captures per second is given by:

$$\phi \sigma N = C_\gamma$$

where

σ = cross section

ϕ = neutron flux (average)

N = number of target atoms

assuming one gamma per capture, the capture gamma dose rate would equal:

$$D (R/hr) = 1.86 \times 10^{-6} E (C_\gamma)$$

However, to find the average neutron flux, the energy dependent transport equation would have to be solved for each section of the shield (use of DOT or MORSE computer codes would be necessary).

Ignoring the capture gamma dose rate, an order of magnitude total dose number can be found by:

$$D_\gamma E (e^{-0.045(x)}) + D_n E (e^{-x/R}) = \text{Total}$$

where

x = thickness

R = Relaxation length in water

Due to the problems associated with the calculation of the capture gamma dose rates the following method was used.

A quick, accurate total dose rate can be calculated using Figures A9-1 and A9-2 for any size WEP shield and any size Cf-252 source. By reading the fast neutron and slow neutron dose rate numbers from Figure A9-1 and adding these numbers together to obtain D_n . Repeat the above process with capture gamma and primary gamma curves (Figure A9-2) to obtain D . Add D and D_n and multiply by the weight of californium in milligrams and by 2.3×10^9 (neutrons/sec-milligram). Divide the resulting number by $4\pi r^2$ to obtain the total dose rate in milliRem/hr.

Figure A9-1

Neutron Dose Rate Curve For WEP and Cf-252

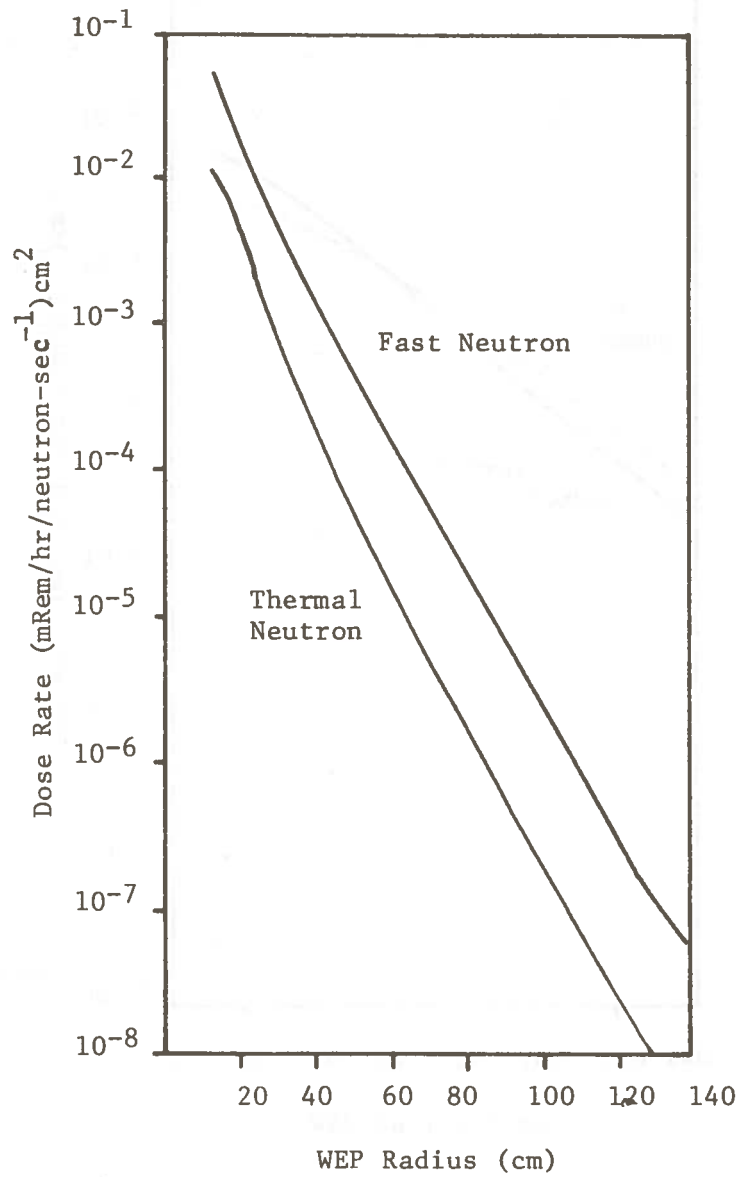
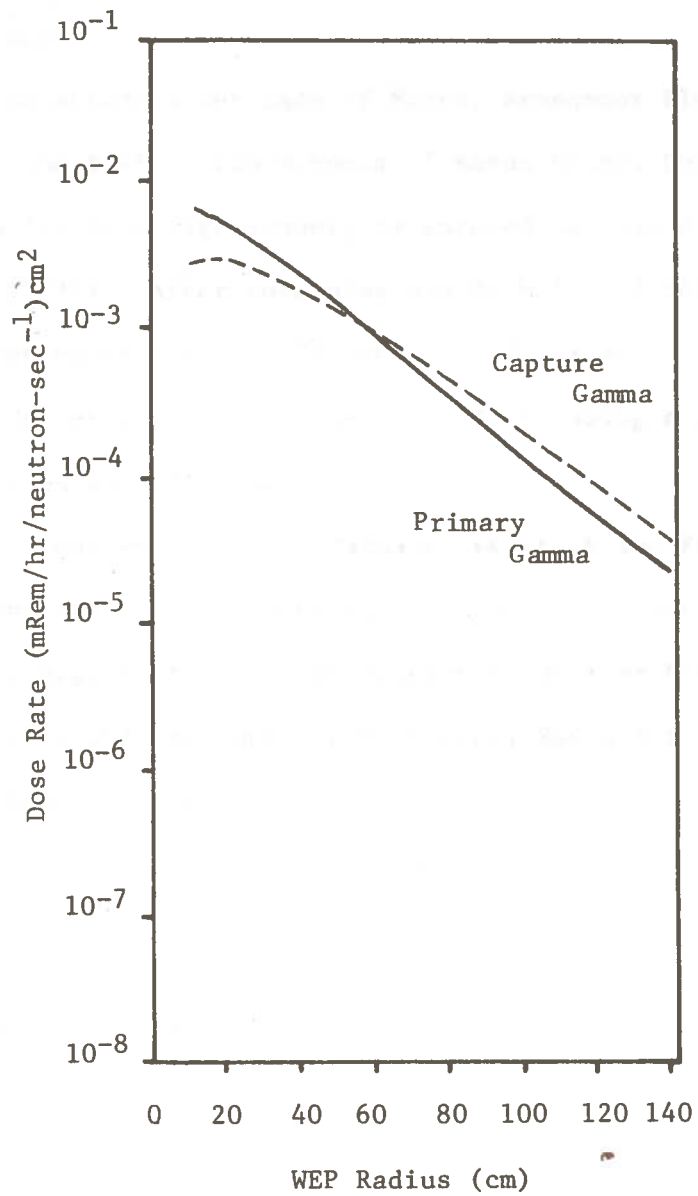


Figure A9-2

Gamma Dose Rate Curve For WEP and Cf-252



VITA

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He attended our Lady of Mercy, Broadmoor Elementary and Broadmoor Junior and Senior High Schools of Baton Rouge, Louisiana. Upon graduation from high school, he entered Louisiana State University in May, 1967. After obtaining his Bachelor of Science in Mechanical Engineering in May of 1971, he entered graduate school of Louisiana State University in September of 1971 pursuing a degree of Master of Science in Nuclear Engineering.

He has worked as a Graduate Assistant and Research Associate for the Department of Engineering Research. At present he is a candidate for the degree of Master of Science in Nuclear Engineering and upon graduation will be employed by Coastal Radiation Services, Inc., of Baton Rouge, Louisiana.