

IN-PLACE DETERMINATION OF CEMENT  
IN CONCRETE BY ACTIVATION ANALYSIS

A THESIS

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The contents of this Thesis reflect the views of the author who is responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the State or the Federal Highway Administration. This Thesis does not constitute a standard, specification, or regulation.

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## ABSTRACT

Thermal neutron activation analysis of cement content in Portland Cement Concrete should provide a reliable method of predicting possible structural failure in certain highway sections. Design of a field portable shield-activation assembly should be based on reliability, effectiveness of biological shielding, and predictability and reproducibility of irradiation geometry. The activation assembly which has been constructed was designed to hold 50 micrograms of californium-252. This system has provided the necessary thermal neutron flux and biological shielding required for the analysis of many laboratory samples to an accuracy of  $\pm 5\%$  (of cement added).

## CHAPTER ONE

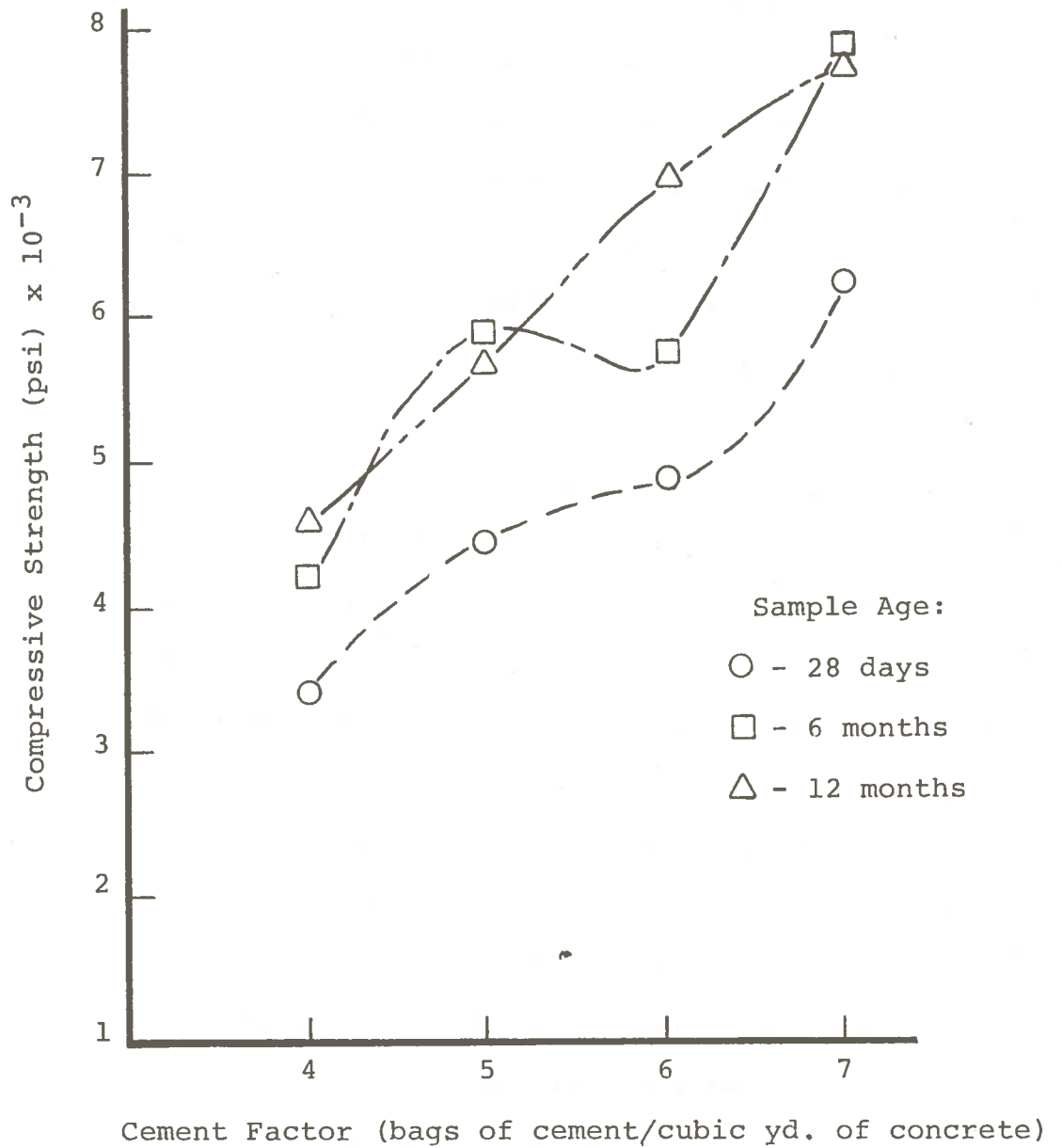
### Introduction

The advantages and rewards of a good quality assurance program have long been known. Whether that program be composed of destructive, or non-destructive techniques, or both, there can be little argument that the investment involved is returned in increased quality of the finished product. When we consider highways in this light, we find that a technique which could predict failures in highways and general types of concrete structures, could be very valuable.

At present, no routine method of analysis is in use which analyzes set concrete surfaces to determine cement content for the purpose of assessing serviceability. Since cement content and its uniformity plays a major role in determining the loading limits of a surfaced area (see Figure 1-1), it is reasonable to assume that an accurate evaluation of cement content could aid in locating areas of potential heavy maintenance.

At present, the accepted method of measurement of cement content of hardened Portland Cement Concrete<sup>1/</sup> involves a long and tedious chemical analysis (Appendix A). The analysis requires a high degree of technical skill, and many

Figure 1-1  
Plot of Concrete Capacity.



thousands of dollars invested in laboratory facilities and equipment.

Some determinations have been performed using atomic absorption spectroscopy, X-ray fluorescence, and gamma ray backscatter; however, these methods are not prescribed as official. Other than the more obvious drawbacks involved in these methods (lack of portability in the case of atomic absorption and X-ray fluorescence) all of the above methods treat non-representative size samples. That is to say that surface conditions of identical samples could cause highly different assays, and vice-versa.

The process of neutron activation analysis, as discussed in the research described herein, should prove to be more accurate, quicker, and easier than the methods described above. Activation analysis provides an in-depth (up to 5 inches into sample) analysis of large quantities (over 3,000 grams) of sample material with accurate results. There is also the added advantage of on-site operation, with proper design considerations. With a portable system, quantitative determination of cement<sup>e</sup> content in concrete to within five percent of the absolute amount of cement added are possible. The resultant data may be made available for plastic concrete in as short a time as sixteen minutes, however, we are considering cement in pavements that are set and so no such short analysis times are necessary.



The process of activation analysis involves producing radioactivity in specimens by radiation bombardment, observing the specific energies of the resultant radiations, and correlating these to obtain a qualitative or quantitative assay. Thermal neutron activation of a specimen is accomplished by subjecting it to a thermal neutron flux for a period of time to be determined when several factors have been evaluated. The neutrons are captured by the nuclei of a variety of atoms in the sample. The capture of these neutrons by a stable atom yields one nuclear transformation which may result in the formation of a radioactive species. The species created emits electromagnetic and/or particulate radiation which is characteristic of that atomic nucleus. The amount of any isotope manufactured in this manner may be expressed by the formula:

$$A = N\phi\delta [1 - \exp(-0.693 t/T)] [\exp(-0.693 t_a/T)]$$

Where:

$A$  = amount of induced activity (disint./sec.)

$N\phi\delta$  = rate of production

$N$  = number of target atoms of material of interest

$\phi$  = neutron flux (n/cm<sup>2</sup>-sec.)

$\delta$  = capture cross section ( $\times 10^{-24}$  cm<sup>2</sup>)

$1 - \exp(-0.693 t/T)$  = saturation factor

$\exp(-0.693 t_a/T)$  = decay factor

$t$  = irradiation time

$t_a$  = decay time

T = half life of the radioisotope of interest

The accepted method of detection and classification of isotopes used in the manner described, is by measuring emissions and providing a visual or tape read-out showing number of emissions versus energy.

Since the isotope of interest in this study is <sup>49</sup>Ca, the gamma ray energies to be observed are 3.08 and 4.05 Mev. Other gamma energies which may be observed are found in Table 1-1.

The relatively high energies of these two radiations provide a wide energy gap between the <sup>49</sup>Ca isotope and the most energetic emissions of other isotopes found in irradiated Portland cements. A graphical (energy versus number of events) relationship of the resultant radiation from cement (Portland) irradiation by a thermal neutron flux is presented in Figure 1-2.

The activation analysis technique used in this study will be to design a field portable system capable of determining, to a high degree of accuracy, the percent cement in a particular mix of Portland cement. In order to make the system as simple to operate as possible, the data obtained must be in a most usable form. The output from the equipment to be used will be nothing more than a number that, once transferred to a calibration curve (Fig. 1-3) may be read directly as cement content (bags

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	<sup>28</sup> Al	0.234 b	2.3 min.	1.78
Calcium	<sup>47</sup> Ca	1.2 b	4.7 days	0.81, 1.30
Calcium	<sup>49</sup> Ca	0.2 b	8.8 min.	3.08, 4.05
Iron	<sup>59</sup> Fe	1.2 b	45 days	1.09, 1.29
Oxygen	<sup>19</sup> O	0.0002 b	29 sec.	0.2, 1.36
Potassium	<sup>42</sup> K	1.5 b	12.4 hrs.	1.52
Sodium	<sup>24</sup> Na	0.53 b	14.8 hrs.	1.37, 2.25
Sulfur	<sup>37</sup> S	0.014 b	5.1 min.	3.1

\*Thermal neutron activation cross section  
in barns ( $1 \text{ b} = 10^{-24} \text{ cm}^2$ )

of cement per cubic yard). The analysis method discussed herein will not only provide a cement percent content result of within a few percent of amount, but will also provide a method which does not require the removal of any sample to a laboratory. The accuracy mentioned is far better than that available with presently prescribed methods, and is also quicker and less expensive per sample.

Figure 1-2  
Concrete  
Gamma Spectrum

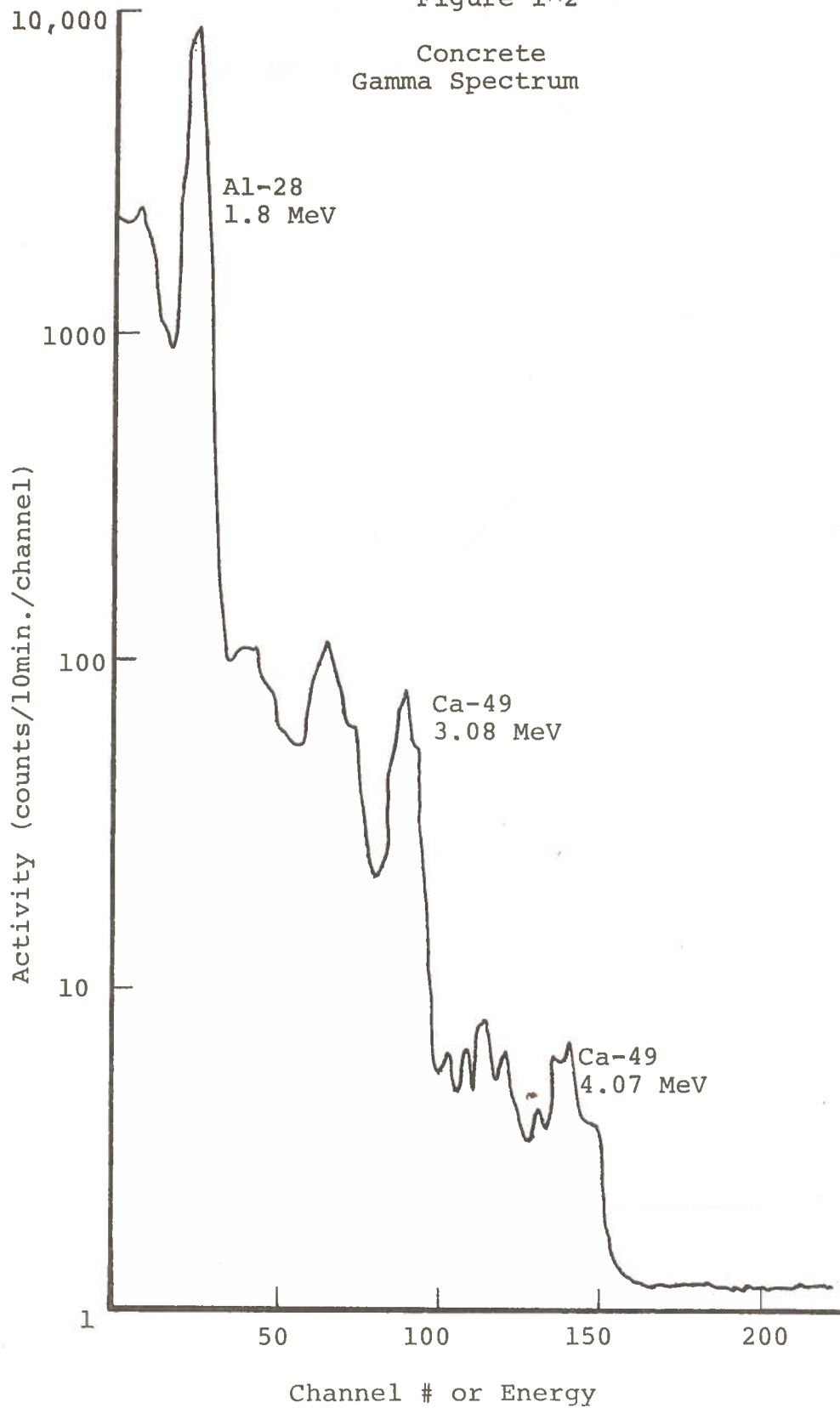
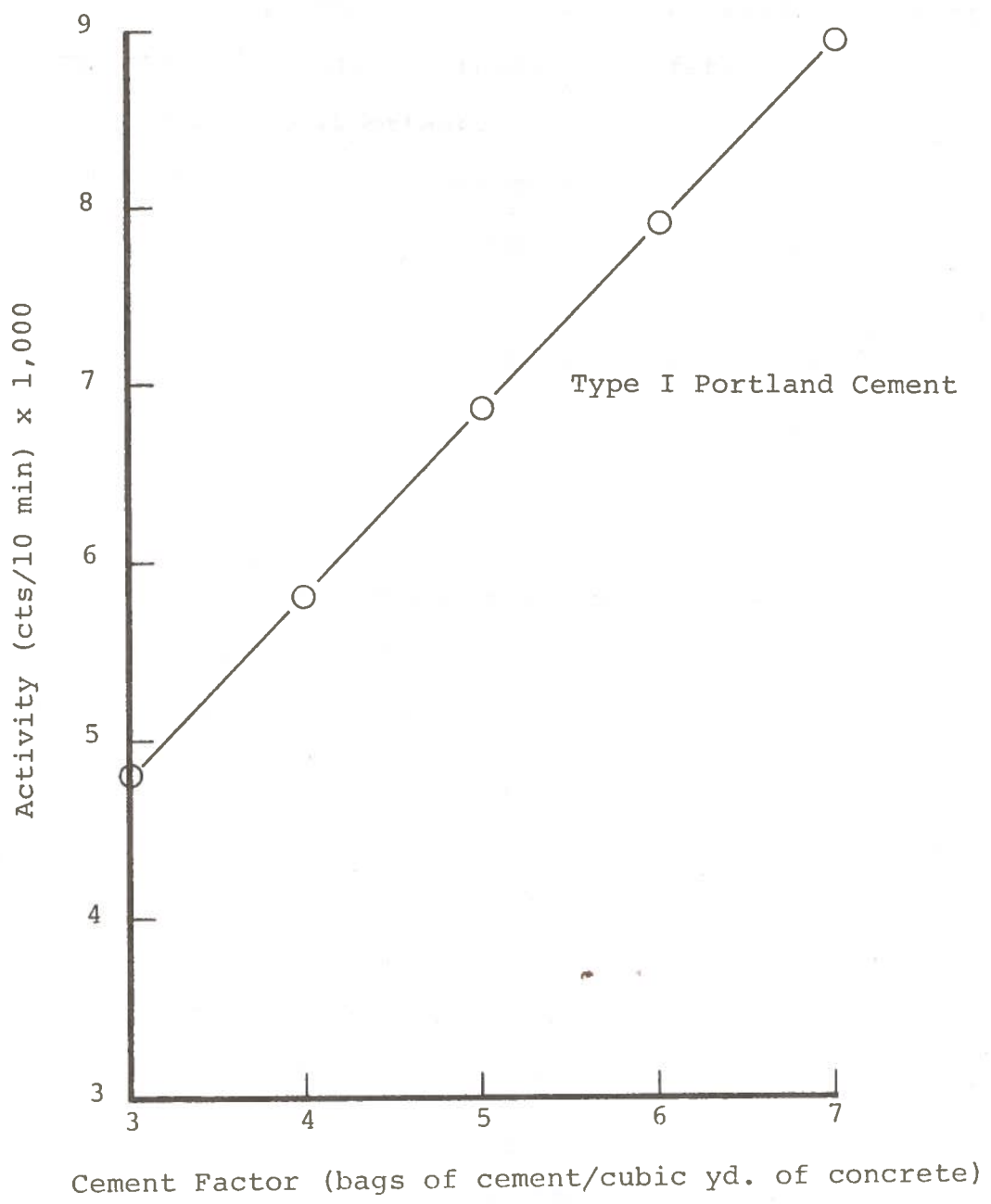


Figure 1-3  
Sample Calibration Curve



## CHAPTER TWO

### Design Criteria

Reliability and durability are not the only requirements for the apparatus to be used in the study of cement in concrete. Portability, radiation safety, and ease of operation are also important.

In other words, the design must:

1. Be a reliable and reproducible supply of neutrons.
2. Be safe to work in the vicinity of for a reasonable amount of time, presenting no radiation hazard to operators or support personnel.
3. Present a reproducible geometry for activation.
4. Possess a stable and reliable counting system.

A reliable and reproducible neutron supply may be achieved with an isotopic source. Neutron generators offer an alternative to isotopes, however, they require a well regulated power supply for the high voltage D.C. supply needed for operation. There is also a problem with reproducing flux levels from such machines. This may be accomplished by using an absolute calibrated  $\text{BF}_3$  detector for direct measurements of the thermalized neutron flux, or indirect measurements using such detectors as Pilot-B crystals fitted to photomultiplier tubes. Both

of these systems require costly, sophisticated equipment solely for flux determination. Even though the neutron flux is monitored, there is still the problem of operating the neutron generator so that the sample-to-sample neutron irradiations are constant.

Radioisotopes, on the other hand, supply a predictable flux which may be determined by relating the age of the source to its half-life plot. Half-life is a measure of the rate at which a radioactive nuclide gives up its characteristic radiation as it spontaneously changes its configuration. For our use, the characteristic radiation desired is neutrons, and a long half-life is desirable. The long half-life would allow for the use of a source for a reasonable length of time. The nuclide  $^{252}\text{Cf}$  undergoes spontaneous fission as one of its decay methods, resulting in a ready supply of fission energy spectrum neutrons. The half-life of this radioisotope is 2.65 years. Composite sources, PuBe, AmBe, etc. give long half-lives, but low neutron fluxes (see Table 2-1).

Then too, there is the fact that an isotope can not be "turned off", as a particle accelerator can, resulting in a rather large biological shield required for transport and storage of the isotopic source.

For this particular design, a source of Californium was selected. The particular isotope in question,  $^{252}\text{Cf}$ , undergoes a spontaneous fission resulting in the emission of 3.76 neutrons/fission in a spectrum having an average

Table 2-1

ISOTOPIC NEUTRON SOURCES

Type	Half-Life	Neutron Yield (n/sec-mg)	Gamma Exposure Rate (R/hr at 1 meter)	Heat Generated (Watts)	Volume (cc)
<u>Alpha-Neutron</u>					
241Am-Be	458 years	$8 \times 10^3$	2.5	750	22,000
238Pu-Be	89 years	$5 \times 10^4$	0.4	550	350
239Pu-Be	24,400 years	100	3.75	500	242,000
210Po-Be	138 days	$1 \times 10^7$	2	640	200
242Cm-Be	163 days	$8 \times 10^4$	0.3	600	2
<u>Photo-Neutron</u>					
124Sb-Be	60 days	$1.9 \times 10^5$ *	45,000	20	200
<u>Spontaneous Fission</u>					
252Cf	2.65 years	$2.34 \times 10^9$	2.9	0.8	1

These Parameters Normalized to a Yield of  $5 \times 10^{10}$  n/sec

Gamma Exposure Rate Heat Generated Volume (cc)

\* Yield in n/sec-ci of 124Sb.



energy of 2.3 MeV, with a most probable energy of 0.8 MeV. An alpha emission is also characteristic of  $^{252}\text{Cf}$ , occurring approximately 31 times for every fission produced. Low energy gamma rays from the fission process are also emitted from  $^{252}\text{Cf}$  at a rate of  $1.3 \times 10^{13}$  photons/sec-gm.

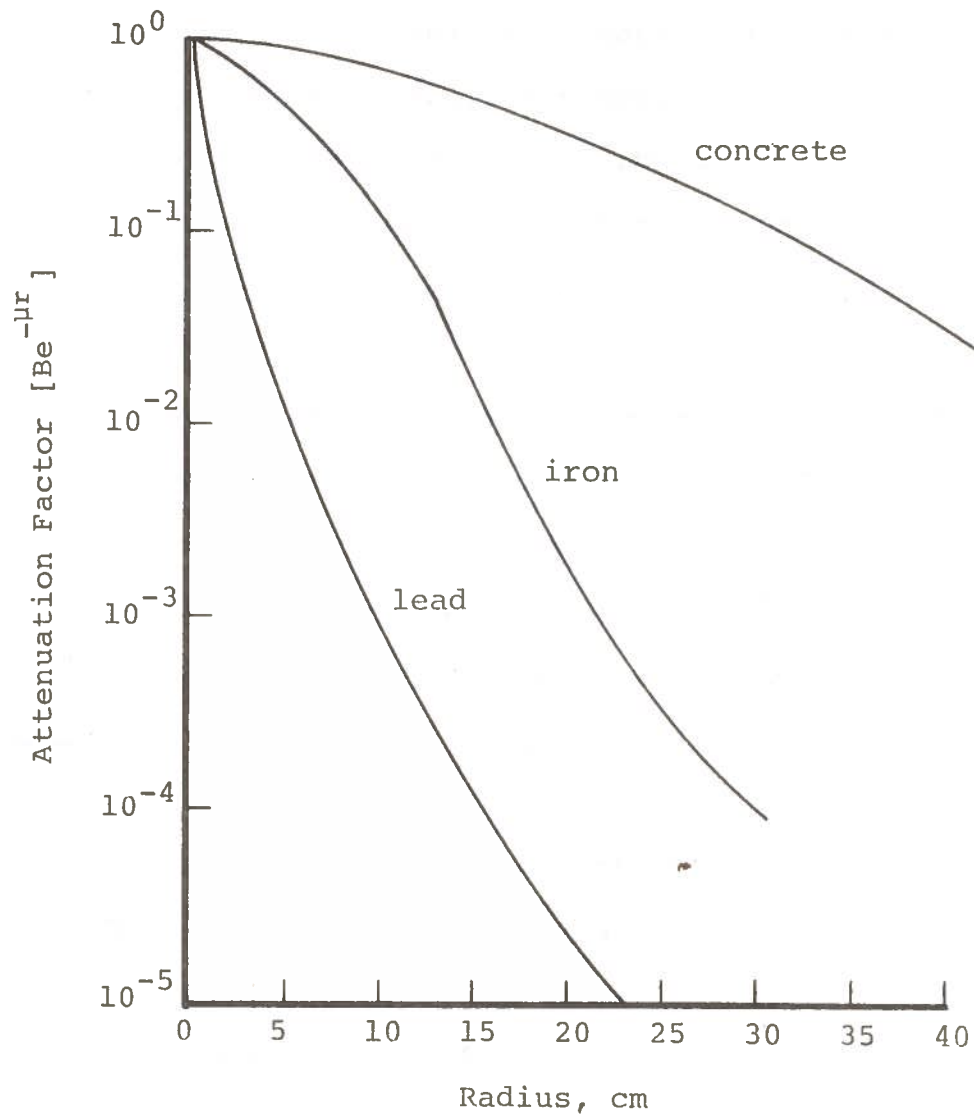
The radiations described above must be shielded against for the protection of technicians and others involved in work in adjacent areas to those used for storage and determination of cement. The alpha radiation will prove no problem because of its short range, and therefore ease of shielding. The gamma photons are more of a problem and require a primary gamma shield of some thickness. Knowing the size of the source to be used in the determination, a value may be taken from Figure 2-1 giving thickness of lead required to produce a given attenuation to primary gammas.

The neutrons from the  $^{252}\text{Cf}$  present both the most significant biological hazard, and the most difficult radiation shielding problem. The neutrons present a three-fold problem to be reckoned with in shield design.

1. Fast neutrons, many of which may be slowed in the shield material and subsequently captured by poisons purposely introduced for that purpose. All of these will not be stopped however, and will represent a major portion of that dose delivered to the technicians.
2. Thermal neutrons, which were fast and have been

Figure 2-1

Attenuation of Primary Gamma Rays  
Savannah River Laboratory Report DP - 1246



thermalized close to the shield wall and subsequently escaped before capture. These contribute relatively little to total personnel dose accumulated.

3. Secondary gammas are the other major contributor to accumulated personnel dose. These gammas are the price paid for slowing down the fast neutrons by inelastic scattering and by the capture of the low-energy neutrons. The capture of thermal neutrons by hydrogen is the principle source of secondary gammas. As their energy is 2.23 MeV, these gammas are rather penetrating. Since these occur outside the primary gamma shield, it is of no use in attenuating secondary gammas, unless of course, the lead shield is positioned on the outside of the neutron shield.

With these considerations in mind, a design must also incorporate previously discussed criteria. A reasonable size to be handled by one or two men, and ease of fabrication are all secondary considerations to biological shielding, but nonetheless important.

The possible shapes to be used:

$$\text{Cube } V = s^3$$

$$\text{Cylinder } V = 0.785 s^3$$

$$\text{Sphere } V = 0.523 s^3$$

Where

$$V = \text{total volume}$$

S = diameter, height, length, or width

As is evident from the preceding formulas, a spherical shape offers the optimum volume-to-weight ratio. A sphere, unfortunately, offers a costly shape to fabricate and would require too much time to engineer into a suitable cask-irradiator.

The cylinder, on the other hand, is easily formed with an outer rigid shell, and an inner source transfer assembly. While not as economical on weight versus volume, its' ease of construction is well worth its' choice.

The primary gamma shield is lead, formed into a suitable shape and size to reduce primary gamma levels at the outer shield surface to be acceptable and in line with prescribed health physics requirements.

The neutron shield material is water-extended polyester (WEP) which is a mixture of 60% water and 40% Alpon 661-P resin<sup>2/</sup> with hydrogen peroxide used as a catalyst. WEP presents a favorable medium for the interaction (and subsequent thermalization) of  $^{252}\text{Cf}$  fast neutrons with the hydrogen nuclei abundant in this material.

The physical properties of WEP make it doubly attractive. Unlike frequently used neutron shielding materials (water, paraffin, glycerin and polyethylene).

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<sup>2</sup>

A product of Ashland Chemical Company, Houston, Tex.

WEP, once in its hardened matrix, represents a non-toxic, fire resistant, leakproof, supple and workable, and reasonably inexpensive alternative. It does not freeze, as water does, nor does it require an outer container to prevent loss of material.

The matrix is not totally stable, however, resulting in uptake and discharge of airborne moisture depending on the humidity of the ambient air. This obviously causes the material to "breathe", and thus interfere with any close tolerance movable surfaces machined into it.

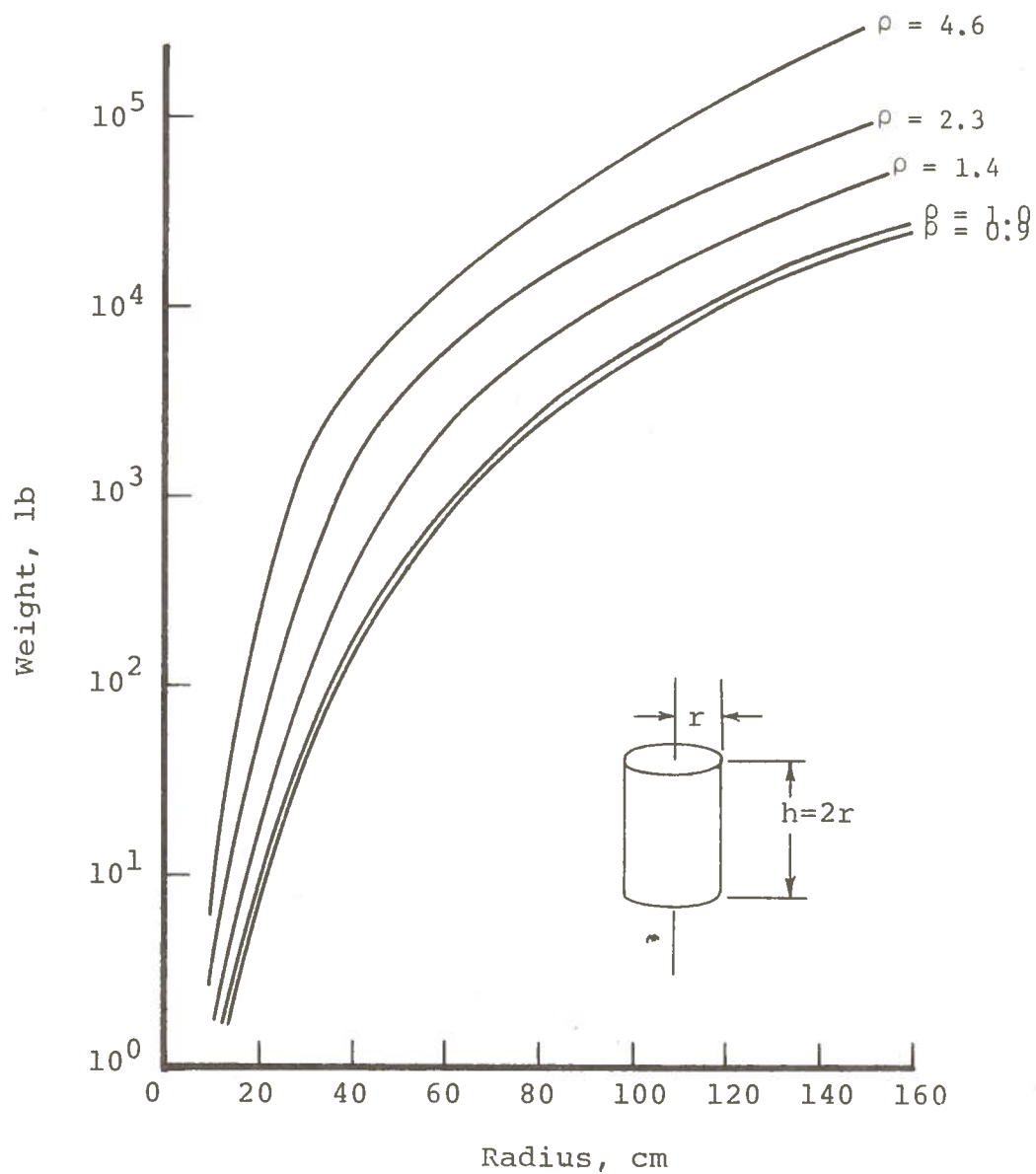
The axle rating of most rental trailers is 1200 pounds, and so the total weight of the activation-shield assembly must be kept at or below this, since the mode of transport to and from the field will probably be in this manner. The relationship of radius-height to weight for a cylindrical shield can be seen in Figure 2-2.

The determinate in the size shield to be used is the source size necessary to achieve the analysis within a "reasonable" time span. In this case, the source to be used is a nominal 37 micrograms of  $^{252}\text{Cf}$ . Exposure rates to personnel using the assembly must not exceed 1.25 rem/calendar quarter (maximum legal exposure to monitored personnel), and it is always advisable to reduce these rates to the lowest practicable.

In this case, the dimensions of the cask-irradiator assembly were chosen to yield a total dose rate on the

Figure 2-2

Weight of Cylindrical Shield as a  
Function of Its Radius for Different Densities



surface of the unit of 100 mrem/hr (gamma and neutron). Based on a forty hour work week, and handling of cask to represent a 1% occupancy factor in an average field of 100 mrem/hr., the average personnel dose should be approximately 480 mrem/calendar quarter.

## CHAPTER THREE

### Preliminary Studies

Since much time had been spent prior to this study on determination of cement content in soil cement, <sup>3/</sup> the use of <sup>49</sup>Ca for such studies was well established. Various sources and sample arrangements were tried. Accelerators were discounted for reasons previously cited. Five curie PuBe isotopic sources did not provide an adequate flux to be useful.

The radioisotope <sup>252</sup>Cf had been used successfully for the measurement of cement content in soil cement mixtures and it became obvious from this experimentation that <sup>252</sup>Cf was probably the only isotopic source that could provide the flux desired in as compact a source volume as possible.

During some preliminary design work, it was decided that an end-on approach to source positioning would be used. All other source arrangements that have come to light in past months have been positioned parallel to the plane of the sample. It was necessary in this case to use a system incorporating a perpendicular source-to-sample

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<sup>3</sup>  
Iddings, F. A., Miller, L. W. Jr., and Pepper, C. E. "A Rapid Field Determination of Cement Content." Proceedings of the 9th Symposium on NDT Evaluation. April 25-27, 1973, San Antonio, Texas.



plane configuration. The reason for this will be amplified later in this section.

A shield assembly was salvaged from a project which had been completed some time ago. This arrangement was modified to accommodate an end-on source arrangement (See Appendix B), and consisted of a WEP filled bucket, drilled through its middle and fitted with a polyethylene "cup" to hold the source at the proper height. A variable thickness plexiglass moderator plate was used between the source and samples.

The use for this arrangement was optimization studies of sample size, moderator thickness, and irradiate-delay-count timing sequences. A source size optimization study was not required, because of the results of previous studies. These studies and experimentation pointed to the use of a source containing about 40 micrograms of  $^{252}\text{Cf}$  as adequate to obtain good results.

Such a source was made available through the cooperation of the Louisiana State University Californium Demonstration Center.

Samples of various size and thickness of ready-mix cement product, and an assortment of Portland Cement Concrete standards, prepared by the Louisiana Department of Highways Cement Laboratory, were used during the preliminary phase.

The activation assembly was positioned behind a concrete block biological shield, stacked to a height of

36 inches. The concrete samples were placed on and removed from the irradiation assembly by reaching over the shield. This method, while being mechanically adequate, presented a possible hazard to that person handling the samples.

The larger samples (11 in. x 11 in. x 5 in.), in particular, required the positioning of the operator's hands to within a few inches of the source for brief periods of time. It was estimated that for such periods the hands would be placed in a 500 mrem/hr. field for approximately 2 seconds per sample exchange. As standard practice throughout this phase of experimentation a tissue-response dosimeter capable of measuring both gamma and neutron dose was affixed to the left hand of that person exchanging the samples.

Operations in the area were coordinated so as to minimize personnel dose, and accurate dosimetry records were kept.

A laboratory counting system was assembled (See Appendix C) using a 5 inch by 5 inch NaI (Tl) detector and associated electronics. The size of the detector was established by research done in this area prior to this time.<sup>3/</sup> Samples were carried to the counting system and placed on the crystal using positioning marks on the top of the support housing. After counting, the samples were returned to the irradiation area, some distance away from the crystal, so as to minimize the background.

Actual dimensions of the field activation assembly

were obtained using the cask mock-up assembly, designed and fabricated in January, 1974. This assembly consisted of a plastic drum, 1/8 in. in thickness with overall dimensions of 50.5 cm. in diameter, and 80 cm. in height.

The purpose of this tank was determination of cask dimensions as affected by boration of WEP shielding material. Five cm. was chosen as optimum thickness for the lead primary gamma shield.<sup>4/</sup> A 10 cm. in diameter casting was poured of lead thought to have as few impurities as possible. The resultant lead cylinder was 10 cm. in diameter by 17 cm. in height. A 3/8 in. hole was drilled into it's vertical axis, to a depth of 10 cm. This then represented the primary gamma shield.

Positioning the primary gamma shield to the middle of the plastic drum was accomplished by use of a pedestal (See Figure 3-1) constructed for that purpose.

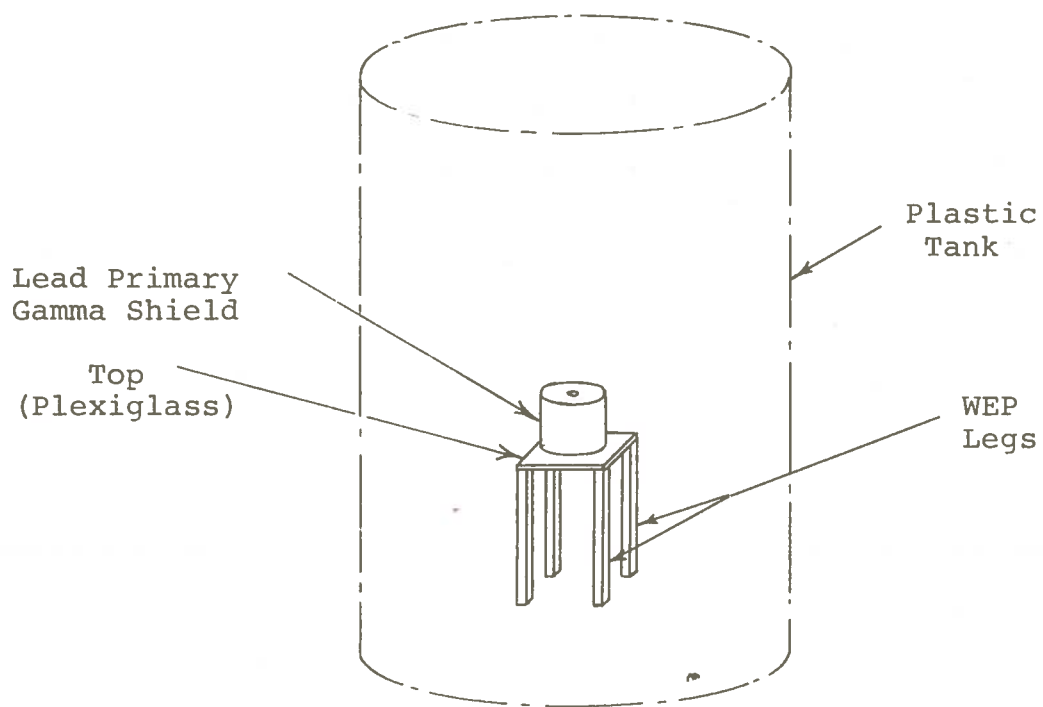
The tank (drum) was filled to 75 cm. of its height with tap water, the proposed  $^{252}\text{Cf}$  source to be used in the field was transferred to it, and placed in the primary gamma shield. Dose rate measurements of both neutron and gamma radiation were made along the outside surface of the tank. Positions of each reading were noted, and the measurements recorded.

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D. H. Stoddard, H. E. Hootman. " $^{252}\text{Cf}$  Shielding Guide". Savannah River Laboratory Report DP-1246. March, 1971, Aiken, South Carolina.

Figure 3-1  
Pedestal and Primary Gamma Shield Assemblies  
Used in Preliminary Study



Boration of the water in the tank was accomplished by use of boric acid. Ten liter amounts of water were removed from the tank and replaced with ten liters of boric acid solution of correct concentration to attain the next desired level of boration. This method proved very efficient provided the ten liters of water used to dissolve the boric acid in crystallin form was 75° C or higher.

Borated water solutions of 1,2,3, and 4 grams per liter were achieved, and dose rate measurements for a number of different positions along the tank axis for each boration level were made and recorded. The information gathered using this apparatus proved most useful in deciding dimensions for the field assembly.

## CHAPTER FOUR

### Results

In a period of just under four months, a final field assembly was tested and deemed reliable to provide a reproducible geometry and neutron flux suitable for cement determination in concrete. Through early successes of preliminary studies, the final design required no modification.

The early experiments with the WEP filled bucket showed the necessary sample size (Figure 4-1) to be in excess of 400 cubic inches. Also from this arrangement it was learned that a 1/4 in. thick end plate would be feasible for the final assembly, as would a 10-2-10 minute activate-delay-count scheme.

The plastic tank experiment, discussed in Chapter Three, resulted in data suggesting a 3 gram/liter boration level (See Figure 4-2) in the Borated-WEP section of the cask. 50 centimeters in diameter and 75 cm. in height were the dimensions arrived at for the cylindrical cask.

The field irradiator (See Appendix D) consisted of a steel outer casing, filled to two-thirds of its depth with B-WEP. The bottom third, the activation section, was filled with non-borated WEP. During the pouring process a canvas reinforced four foot section of 3/8 in. O. D. vacuum tubing was used to form a source transfer

Figure 4-1  
Effect of Sample Size on Resultant Count Rate

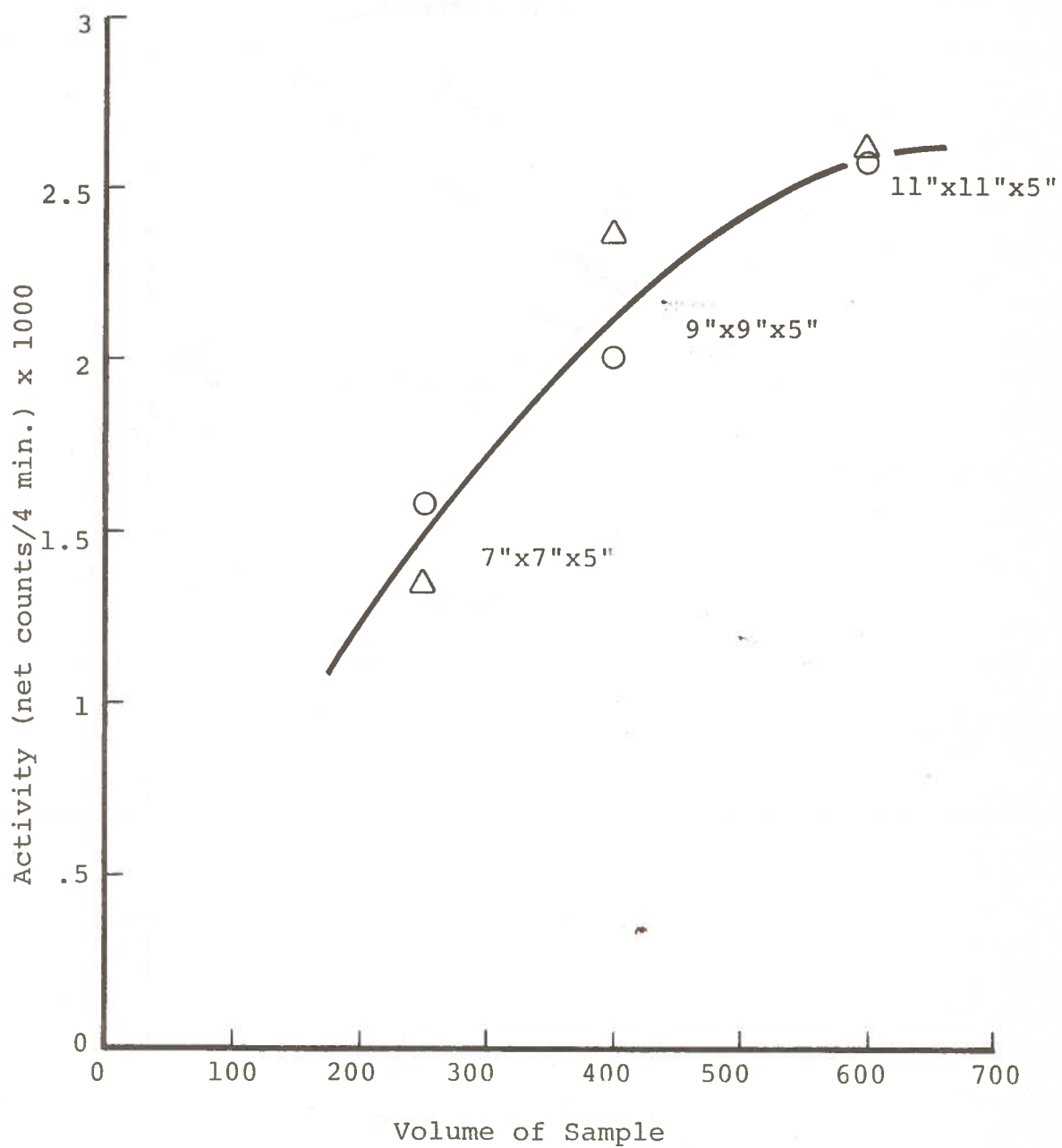
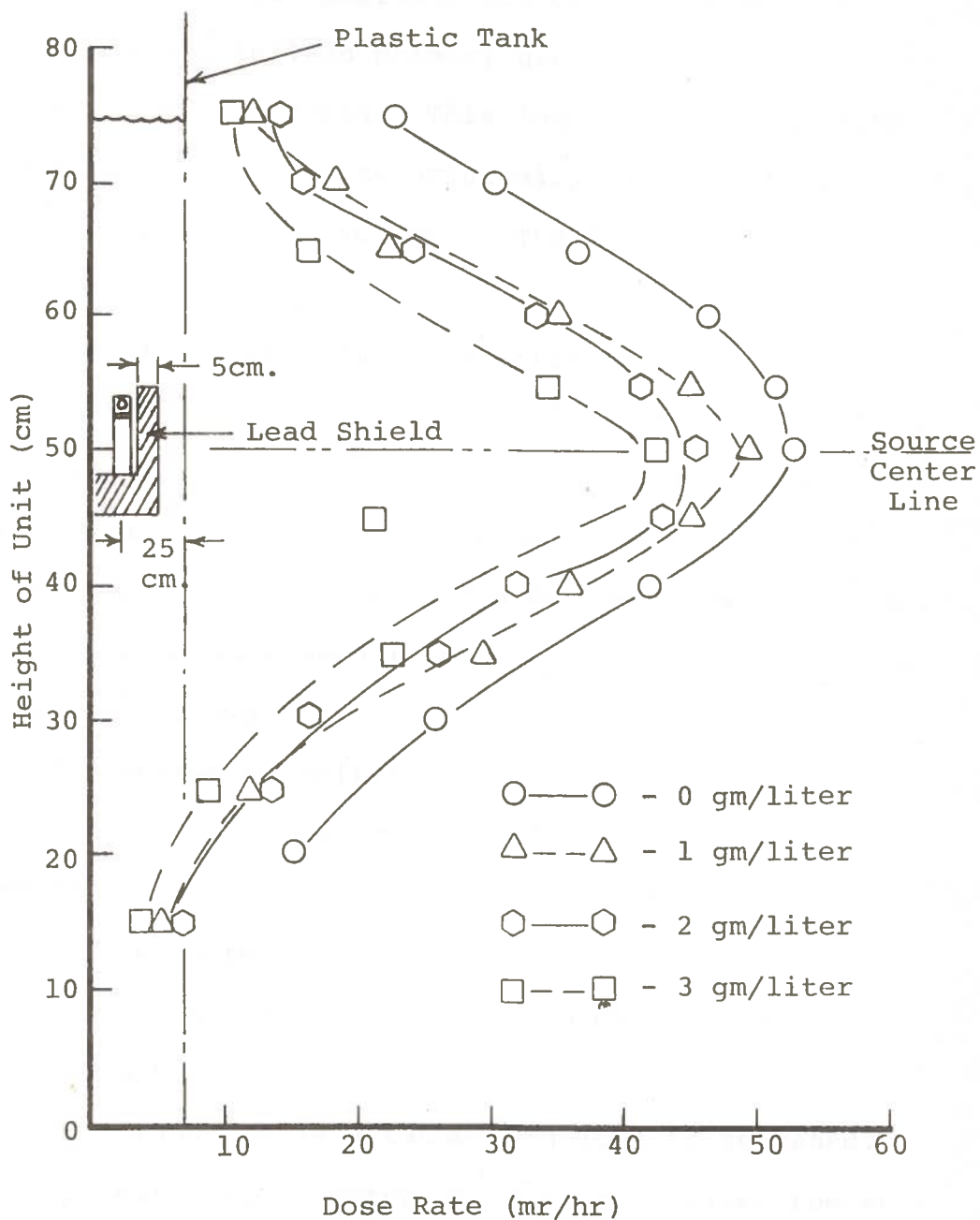


Figure 4-2  
Dose Rate Depression By Boration





tube from the "shielded" to the "activate" sections of the cask. This channel remained after the tubing was pulled out of the cured WEP.

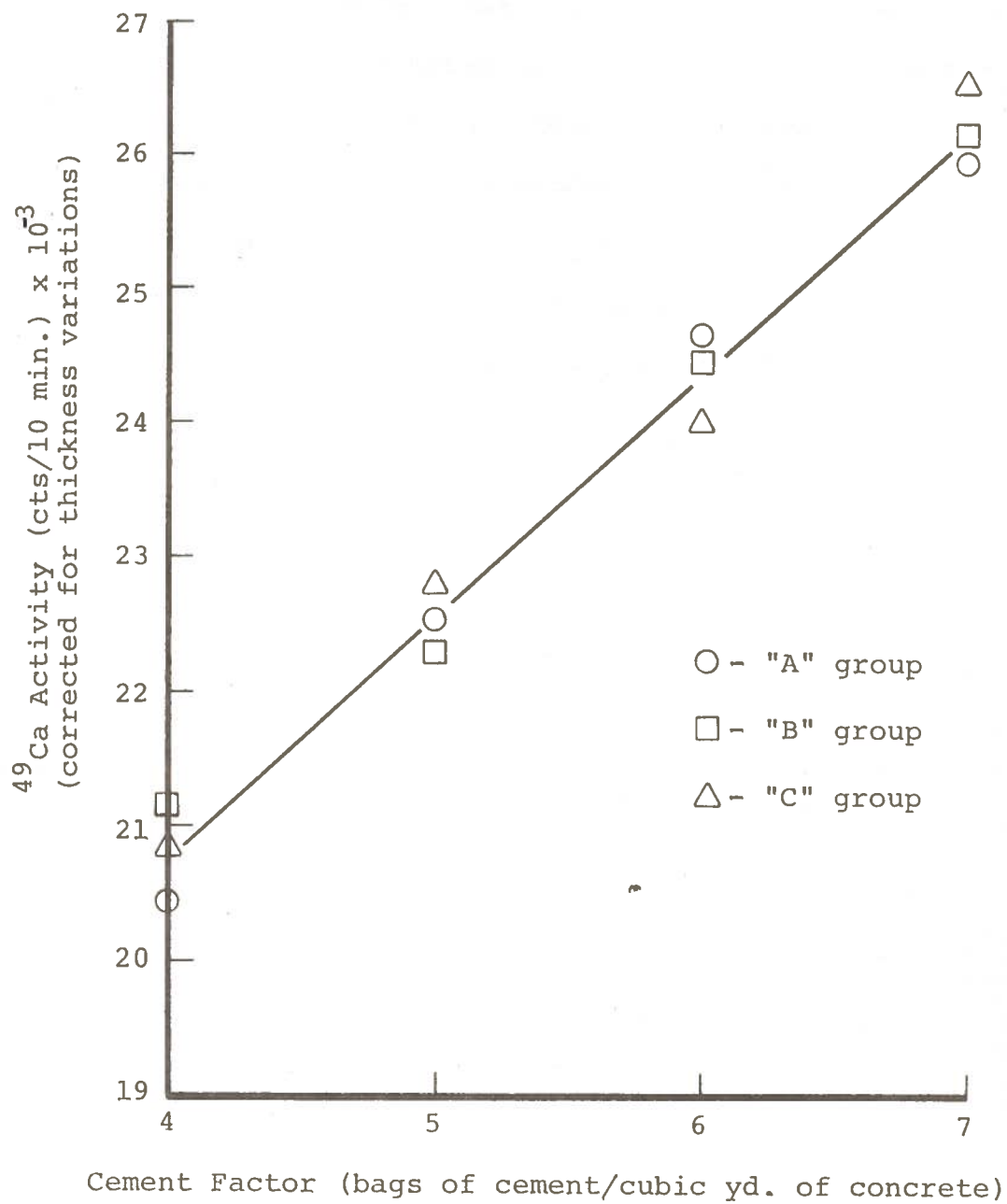
The "shielded" position not only included borated WEP, but also the lead primary gamma shield used in one of the preliminary studies. This lead cask was modified from the cylinder it was originally cast as, to a "C" shaped cask (See Appendix E). The "C" shape eliminated streaming.

Results of the standards irradiated with the assembly, and counted with the field portable counting system (See Appendix F), are shown in Figure 4-3. This calibration curve clearly relates differences in cement content in the very well controlled standards used (See Appendix G).

The procedure used in producing the data in Figure 4-3 is as follows:

1. System is rolled to a position at the edge of the 22" x 22" concrete slab and up-ended so that the moderator end is against the slab. System is centered on the slab.
2. Circumference of system is outlined on the slab with chalk.
3. Crank-out is attached and lock is released.
4. With survey meter ON, operator cranks the source to activate position while standing as remotely to the system as possible.
5. After ten minutes of activation, the source is

Figure 4-3  
Sample Calibration Curve



retracted, cable is released after system is locked, and the irradiator removed to as remote an area as possible.

6. The detection crystal shield assembly is concentrically located on the activated slab position using a template matching the two systems.
7. Exactly two minutes of decay after end of activation, a ten minute count is started. The count includes data produced by gammas above 2.5 MeV.
8. Data were corrected for variations in sample thickness using the curve, Appendix H.

## CHAPTER FIVE

### Conclusions

From the results of the experiments and tests run during this research, it is evident that the system functions as well as originally hoped for. Not only is it possible to determine cement content in Portland Cement Concrete (type 1) to an accuracy of  $\pm 5\%$  of amount added, but the system is also sensitive enough to show differences in mixing procedure in identical batches. The  $\pm 5\%$  accuracy figure is well within the available limits of accepted highway department mixing and preparation procedures.

The cylindrical shield dimensions of 50 cm. in diameter and 75 cm. in height, more than adequately shield operational personnel (See Appendix I) from radiation exposure.

Simple, rapid, and accurate cement determinations can now be performed by relatively unskilled personnel trained in the techniques of radiation safety. By using calibrations for various aggregate ratios, reinforcement type and quantity, and other related variables, the determination of cement in highways may be made more routine.

As a result of observations during this program, it is recommended that a properly designed trailer be procured for the transport of the assembly. Also, that a

cap be fabricated for the moderator end to facilitate activation of cores. Studies should be implemented to obtain data on effects on count rates by aggregate ratio, sub-surface conditions, admixture content, surface glazing by calcium chloride, and other such highway construction and/or maintenance practices.

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Savannah River Laboratory Report DP-1246. March,  
1971, Aiken, South Carolina.

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Concrete." Highway Research Bulletin. No. 340, 1962,  
pp. 118-130.

## APPENDIX A

### Accepted Method of Determination of Cement in Set Concrete As Prescribed In ASTM Standards, Part 10.

Specimens of 10 lb. each are selected, to avoid all but slight inequalities of the concrete mix. The portions are broken down to 2 to 3-in. size pieces with a hammer and 1500 to 2500-g composite samples are dried at 105°C to constant weight.

The samples are heat treated in a furnace to 550°C for three hours to destroy the strength of the concrete. An accurate weight is taken of the sample at this point in the process for use in future equations. The samples are next worked by hand over a No.6 sieve with a pan below, being careful not to lose any sample.

The fraction retained on the No.6 sieve, after weighing, is placed in a beaker and washed with dilute 1:9 HCl. This is followed by several water washes, carefully decanting after each wash. The washed aggregate is dried and reweighed. That material which passes the No.6 sieve is pulverized to pass a No. 200 sieve for the chemical determinations of the acid soluble  $\text{SiO}_2$  or  $\text{CaO}$ .

A 100 g sample of that portion of aggregate that did not pass the No.6 sieve is prepared as above. The combined aggregates are the tested and reported on either the soluble silica or soluble calcium oxide basis,

depending on their response to the acid treatment mentioned above. The soluble calcium oxide basis is used when the aggregate contains substantial amounts of material that yields silica ( $\text{SiO}_2$ ), and soluble silica basis is used when the result is abundant in calcium oxide ( $\text{CaO}$ ).

Soluble silica procedure requires mixing 2-g of the aggregate with 100 ml of HCl (1:3), allowing the mixture to digest on a steam bath for 15 minutes, then decantation (twice) through filter paper for fine precipitates. The resultant filtrate will contain the silica in the form of silicic acids, either in true solution or in suspension in the hydrochloric acid medium.

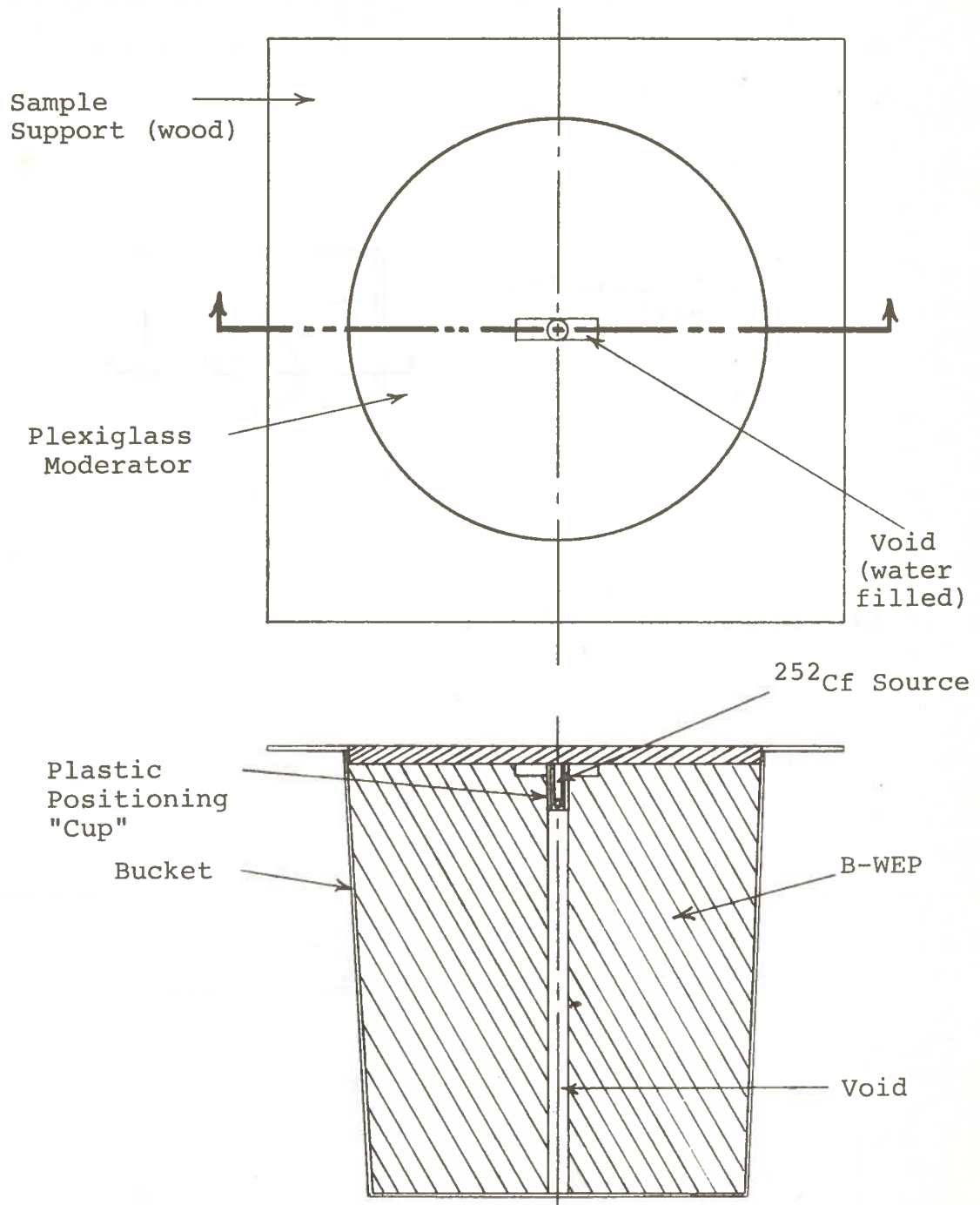
Where the original sample contains substantial calcium oxide ( $\text{CaO}$ ) on acid treatment, 10 ml of HCl (sp gr 1.19) are added to the solution. After several repeats of this, accompanied by evaporation and filtration, the final washings should be free of chlorides and a silica analysis may be made using an HF and  $\text{H}_2\text{SO}_4$  treatment.

The soluble calcium oxide analysis is exactly the same with the exception of the HF and  $\text{H}_2\text{SO}_4$  treatment. The remaining determination is then carried out as that of limestone, quicklime, or hydrated lime.



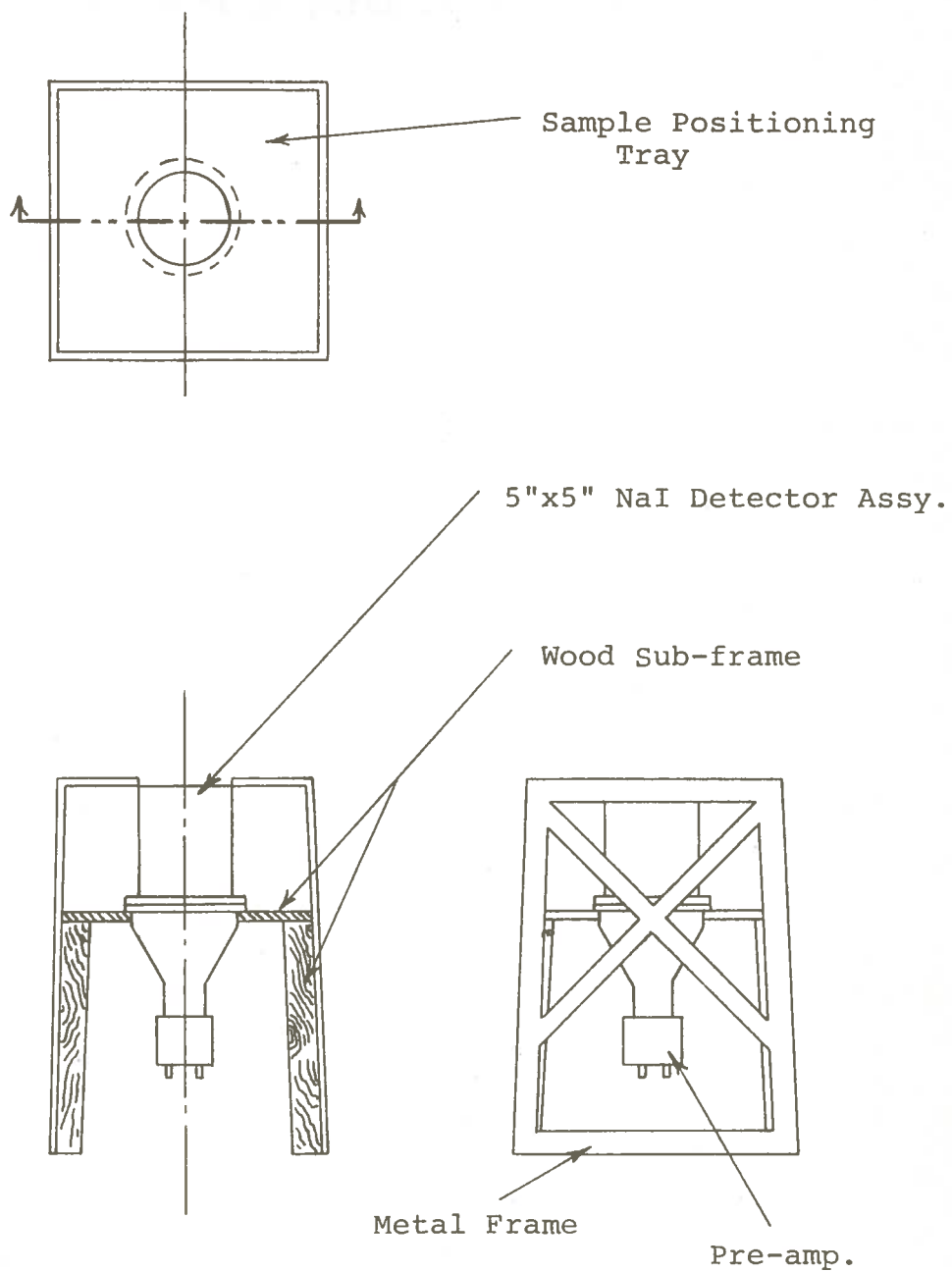
## APPENDIX B

## Laboratory Irradiation System



## APPENDIX C

## Laboratory Counting System



## APPENDIX D

## Field Irradiation Assembly

Two drawings are presented here showing the outer appearance of the field cask/irradiator assembly (Figure D-1), and a full section (Figure D-2) representing the internal arrangement of parts in the assembly.

Figure D-1  
Field Irradiation Assembly

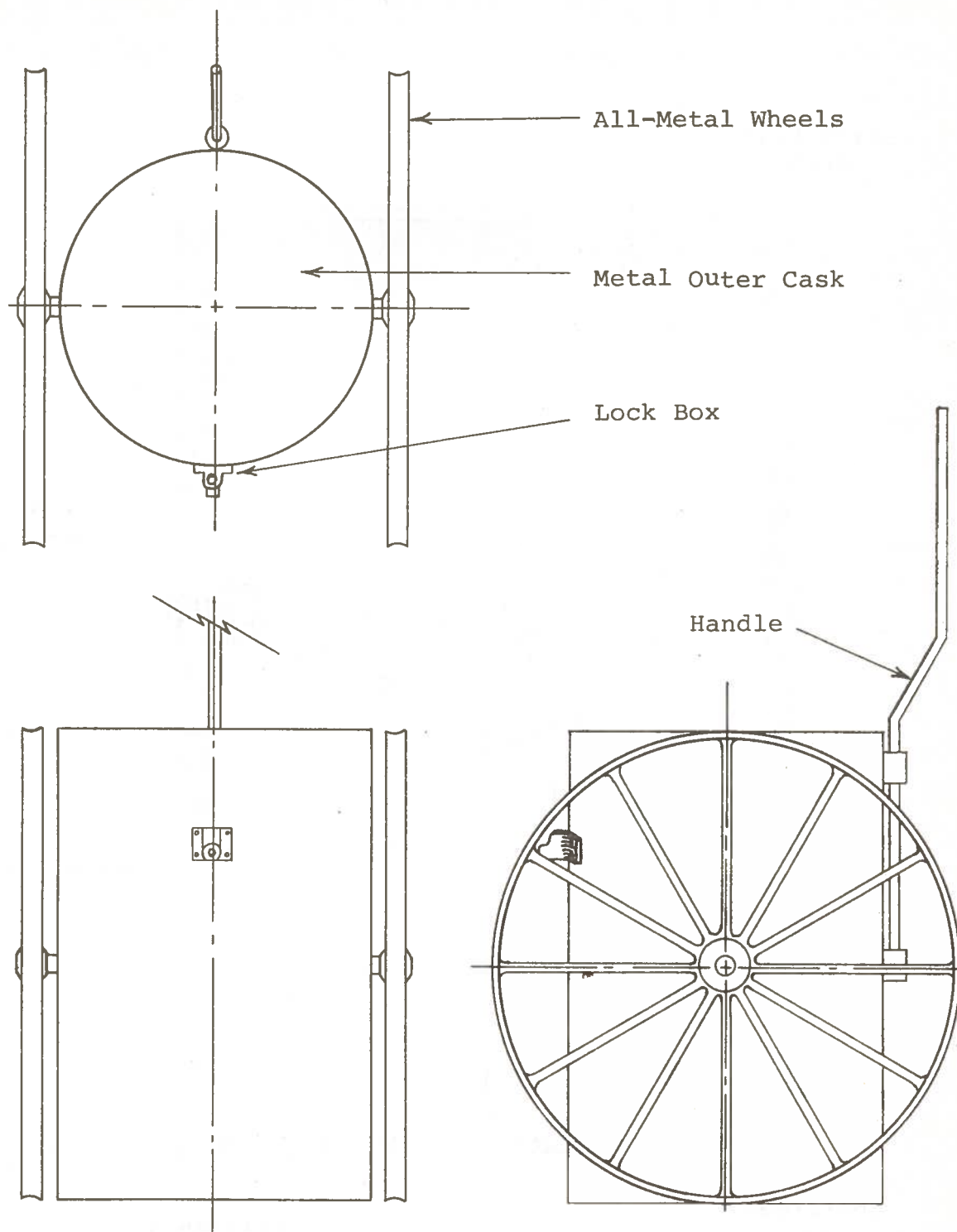
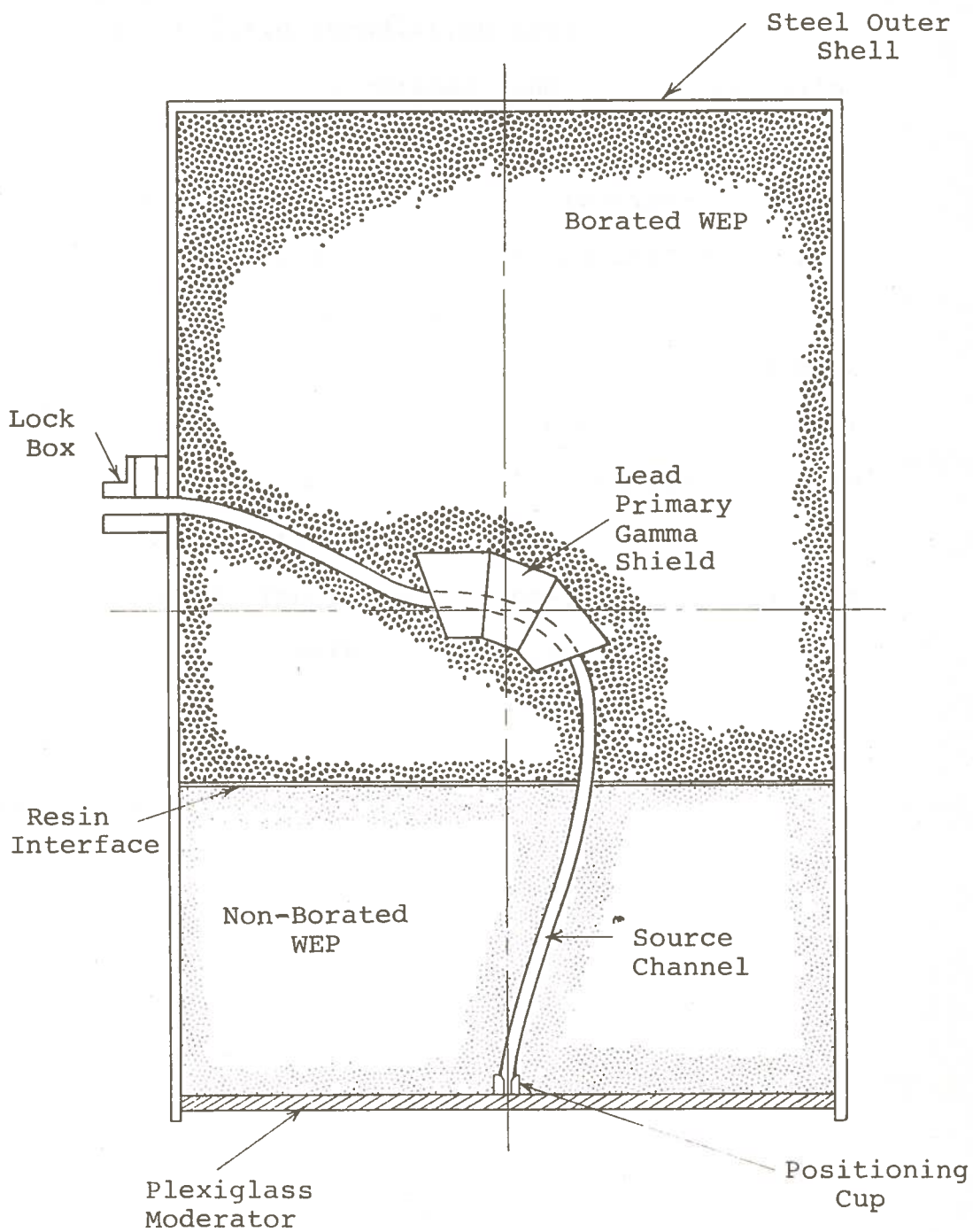


Figure D-2

## Cask/Irradiator Assembly in Cross Section



## APPENDIX E

## Lead Cask Construction

A drawing is presented showing in steps (Figure E-1) the method of construction of the primary gamma shield used in the field irradiation assembly.

Step 1. A cylindrical lead pig is cast with desired dimensions.

Step 2. The lead cylinder is marked with 2  $30^\circ$  diagonal lines along its' axis, and cut into three sections.

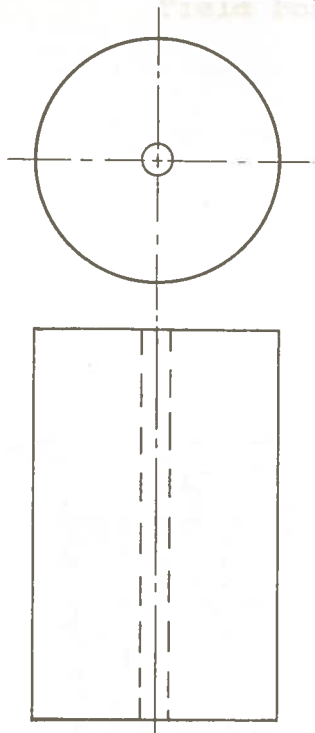
Step 3. The line-of-cut edges are beveled, and the two end pieces are repositioned to the middle section  $180^\circ$  to their original orientation.

Step 4. The three sections, thus joined, are mated together by welding to form as solid a unit as possible.

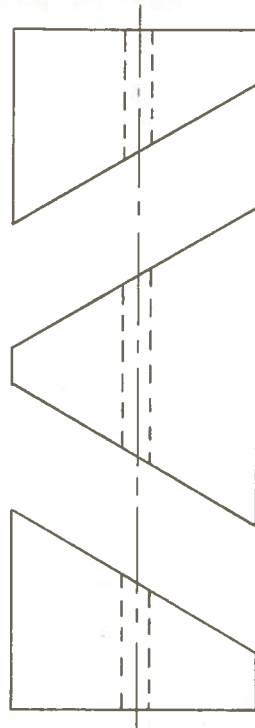
Figure E-1

Lead Cask Construction

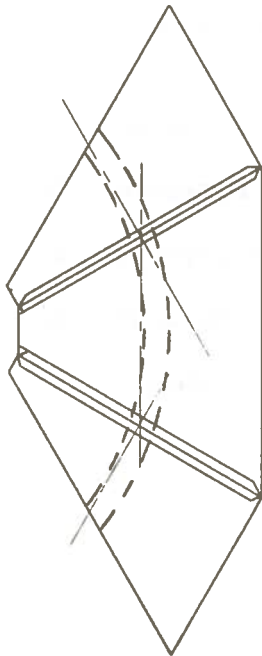
Step 1.



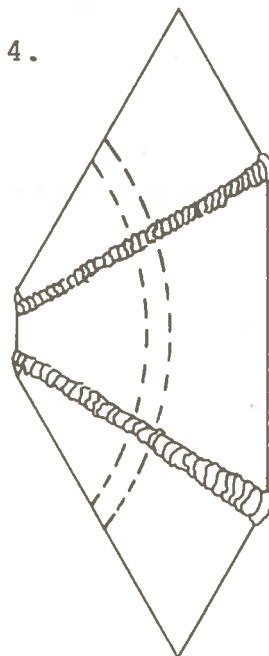
Step 2.



Step 3.

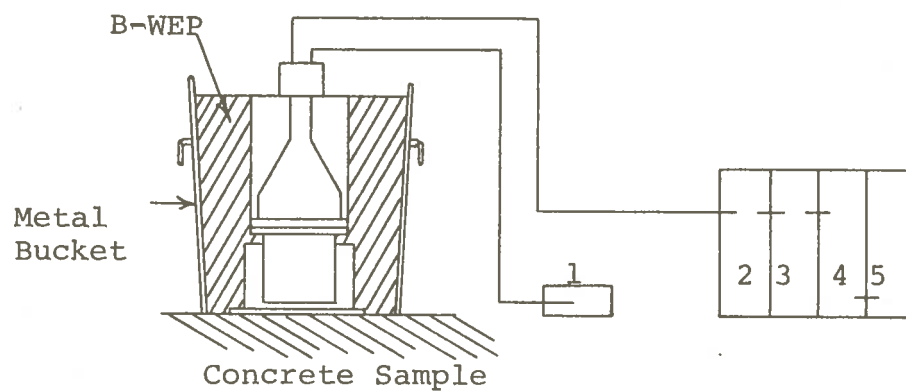


Step 4.



## APPENDIX F

## Field Portable Counting System



- 1 - High Voltage Supply
- 2 - Preamplifier-Amplifier-Discriminator
- 3 - Single Channel Analyzer
- 4 - Scaler
- 5 - Timer



Appendix G  
Calibration Samples

A table is presented here to show the differences existant in those samples prepared to be used in the determination of a calibration curve for cement content in concrete.

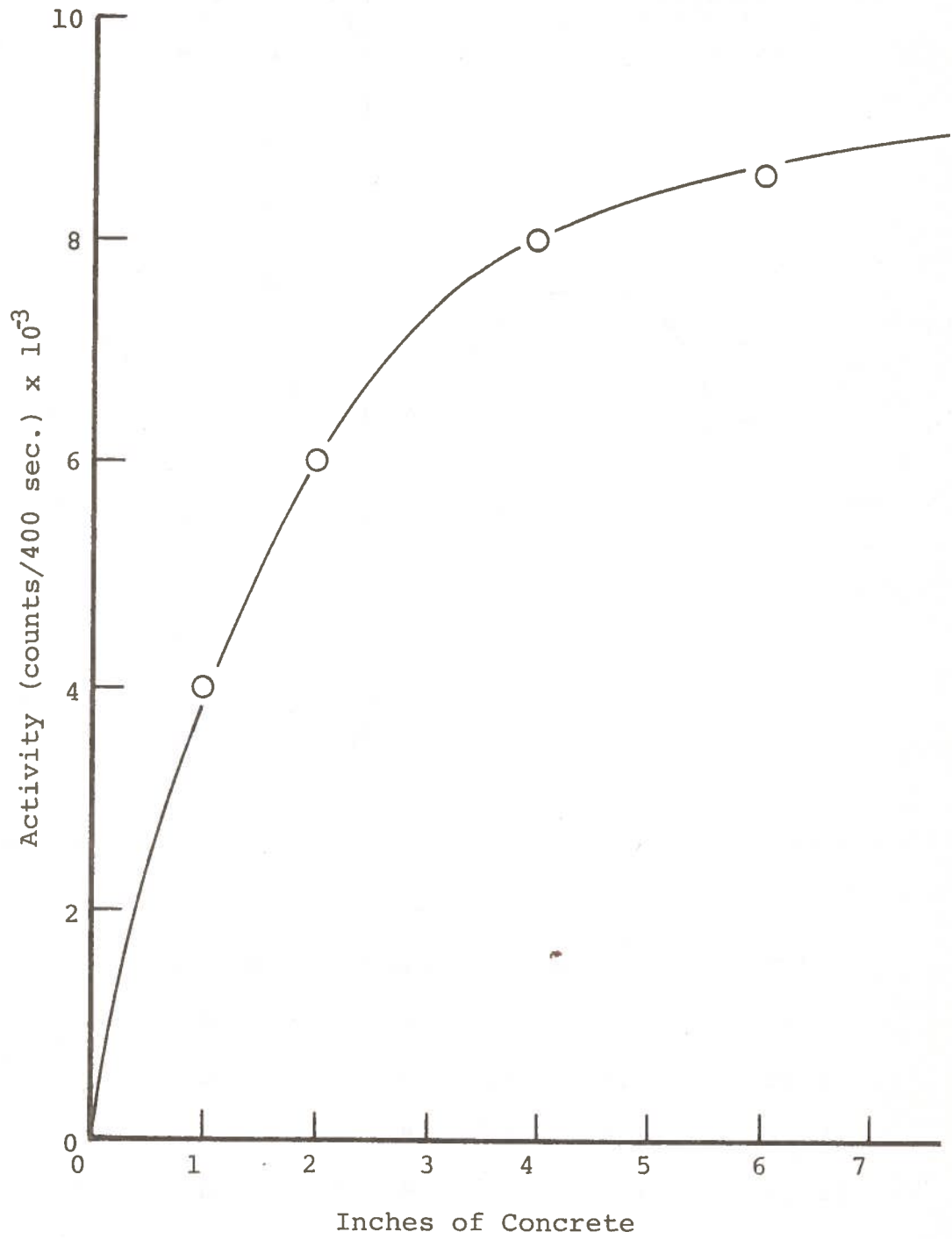
Table G-1  
Calibration Samples

SAMPLE	THICKNESS (inches)	DEPTH/WIDTH (inches)	CEMENT FACTOR*	% CEMENT
1R4-A	4.5	22.25/22	4	10.35
4-B	4.5	22.25/22	4	10.35
4-C	4.5	22.25/22.5	4	10.35
1R5-A	4.0	22.5/22.5	5	13.25
5-B	4.25	22/22	5	13.25
5-C	3.9	22.5/22	5	13.25
1R6-A	4.5	22.25/22	6	15.39
6-B	4.5	21.75/22	6	15.39
6-C	4.5	22/22.5	6	15.39
1R7-A	4.0	22.25/22.25	7	17.50
7-B	4.0	22/22.5	7	17.50
7-C	4.0	22/22.5	7	17.50

\* Bags of cement per cubic yard of concrete.  
NOTE: All samples prepared using 60-40 aggregate ratio;  
Coarse aggregate 60% Fine aggregate 40%.

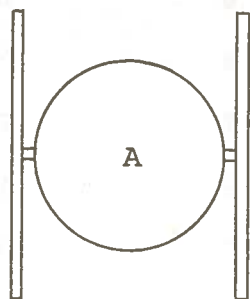
## Appendix H

## Count Rate Versus Sample Thickness

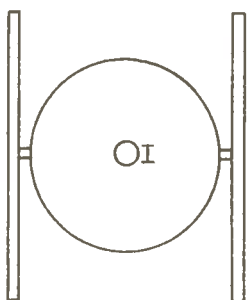
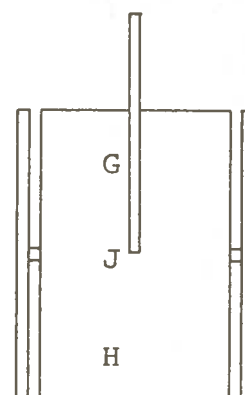
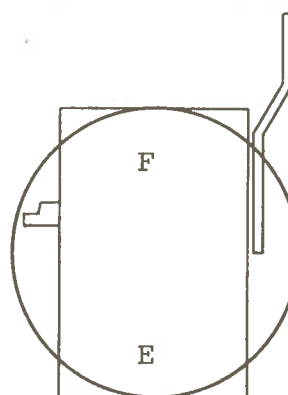
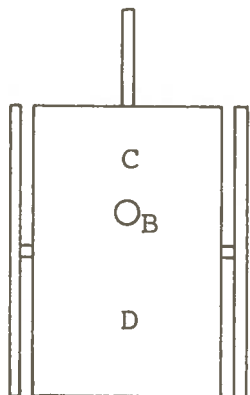


## APPENDIX I

## Survey Report of Loaded Field Assembly



Data taken with 35 micrograms of  $^{252}\text{Cf}$ . in shielded position.



Position	Gamma	Fast N	Dose Rate*		Total
			Thermal N		
A	4	2.6	0.004		6.6
B	6	18.2	0.2		24.4
C	9	13	0.15		22.2
D	6	13	0.13		19.13

Position	<u>Gamma</u>	Dose Rate*		<u>Total</u>
		<u>Fast N</u>	<u>Thermal N</u>	
E	6	10.4	0.13	16.53
F	8	10.4	0.2	18.6
G	7	11.7	0.27	18.97
H	8	11.7	0.2	19.9
I	7	8.0	0.27	15.3
J	12	39.0	0.45	51.45

\* All dose rates given in millirem/hr.

## VITA

Calvin Errol Pepper was born on September 10, 1946, in Franklin, Louisiana.

He attended Jeanerette Elementary, South Cameron High School, Berwick High School, and Morgan City High School. Upon graduation from high school, he enlisted in the Navy and served from June, 1965, to May, 1969. During his enlistment, he was trained as an Aviation Electronics Technician, served aboard several aircraft carriers in various areas of the world, and achieved the final rank of E-5 (Petty Officer Second Class). In the last two years of military service, he served aboard a destroyer in the Viet-Nam war zone for ten months. While attached to the destroyer Robert H. McCard, he received the Viet-Nam service medal, and Viet-Nam campaign medal with stars.

Upon completion of his enlistment, he entered Louisiana State University in May, 1969. After obtaining his Bachelor of Science in Industrial Technology in May, 1972, he entered graduate school of Louisiana State University pursuing a degree of Master of Science in Nuclear Engineering.

He worked as a research assistant 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 Gamma Industries, Inc. of Baton Rouge, Louisiana.

Mr Stule-  
In deepest  
appreciation and highest  
regard - Thank you.

Calvin